CRELL's
CHEMICAL JOURNAL;

GIVING

AN ACCOUNT

OF THE

LATEST DISCOVERIES IN CHEMISTRY,

WITH

EXTRACTS

FROM

VARIOUS FOREIGN TRANSACTIONS:

TRANSLATED FROM THE GERMAN,

WITH

OCCASIONAL ADDITIONS.

VOL. III.

LONDON.

PRINTED FOR R. BALDWIN, PATER-NOSTER ROW.

MDCCXCIII.

LIST

OF

DISSERTATIONS,

CONTAINED IN THIS VOLUME.

ADVERTISEMENT.

INSTEAD of ſending it out in Numbers, it is intended to publiſh this Journal *in future, in Volumes, of which one will appear annually. By this alteration in the mode of publiſhing the work, the communication of new diſcoveries made on the Continent will, it is true, be ſlower; but, on the other hand, the great inconvenience which the reader muſt experience when the diſſertations are printed by parts, in different Numbers, and at different intervals of time, will be avoided.*

Excepting one or two of Mr. Scheele's eſſays, which it was neceſſary to inſert with the reſt, in order to complete the Engliſh tranſlation of his works, the Tranſlators have, conformably to the plan on which they ſet out, excluded diſſertations

tations merely speculative, and confined themselves to such as are of an experimental nature; selecting from among these, such as appeared most interesting on account of the new and remarkable phenomena which they present; such as recommended themselves by their usefulness in the arts, or their application to œconomical purposes; and, lastly, such as might serve as models of chemical investigation to other, and especially to young, experimenters. Among dissertations of the first kind may be reckoned (not to mention any of Scheele's) those of Ruprecht, Westrumb, Tihavsky, Bindheim, Lowitz, Trommsdorf, Fuchs, Wiegleb, &c; among dissertations of the second kind, those of Vogler, Watt, Gadolin, Giobert, Tuchten, Kels, &c; and among dissertations of the last kind, those (not to mention Scheele's) of Pelletier, Klaproth and Hielm.

In the selection of English dissertations the same objects have been kept in view; and our readers need not to be told that the papers of Wedgwood, Keir, Kirwan, and Beddoes are rich in new and interesting facts and observations.

Under

Under the head of Chemical News *is given, along with other occasional articles, some account of the principal memoirs in the Annales de Chimie, and also of the most remarkable dissertations on chemical subjects in the Journal de Physique. If some of Mr. Pelletier's, Berthollet's, &c. late experiments be not therein noticed, it is because a translation of such of their memoirs, at full length, is intended.*

As this work is a translation of only some parts of Dr. Crell's Journal*, *and as it, moreover, contains a large proportion of matter derived from other sources, it is judged improper to retain that Author's name. Accordingly the future volumes will only have for their title* The Chemical Journal. *As before, however, they will, for the most part, consist of papers translated from the German Journal.*

The EDITOR.

March, 1793.

* Under the single word *Journal*, the Translators comprehend the *Annals*, *Supplements*, &c. which may be considered as continuations of the Journal. Dr. Crell's Journal was discontinued in 1781. His Annals were begun in 1784, and have been continued (twelve Numbers yearly) ever since. In the interval between the discontinuance of the Journal and the commencement of the Annals, were published the Newest Discoveries in Chemistry.

I. OF THE PRESENCE OF THE

EARTH OF RHUBARB

IN

VARIOUS KINDS OF VEGETABLES;

By Mr. SCHEELE.

MANY other roots, beſides rhubarb, contain, but in ſmaller quantity, calcareous earth combined with acid of woodſorrel (*calx acetoſellata*). In examining ſuch roots and barks I adopt the following method. I firſt ſlice and bruiſe theſe ſubſtances, then digeſt them in diluted muriatic acid for about a couple of hours, filter off the acid, and drop into it ſome cauſtic ſpirit of ſal ammoniac. If the roots or barks contain any earth of rhubarb, the liquor becomes milky, and the earth falls to the bottom in the courſe of a few hours. In this manner I have detected the preſence of acetoſellated

calcareous earth [oxalited lime] in the following roots and barks.

Roots. Alkanna. Apium. Biſtorta.
Cardopatium (Carlina acaulis)
Curcuma. Dictamnus albus.
Fæniculum. Gentiana rubra.
Hirundinaria (Vincetoxicum)
Lapathum Liquiritia, Mandragora. Ononis. Iris florentina
Iris noſtras. Saponaria. Scilla.
Sigillum Salomonis. Tormentilla.
Valeriana. Zedoaria. Zingiber.

Barks. Berberis. Caſſia fiſtularis.
Canella alba. Cinnamomum.
Caſcarilla. Caſſia caryophyllata.
China (Cinchona officinalis?)
Culilavan. Frangula.
Fraxinus. Quaſſia. Quercus.
Simaruba. Lignum ſanctum.
Ulmus.

II. CHE-

II. CHEMICAL EXAMINATION

OF THE

TERRA PONDEROSA;

By Mr. SCHEELE*.

§. I.

FROM the experiments which I am about to relate it will appear, that the earth contained in ponderous ſpar is not lime, but an earth of a peculiar and diſtinct nature. But before I deſcribe the properties of this earth, it is proper to point out in what manner it may be beſt ſeparated from the heavy ſpar.

§. II.

The ponderous ſpar is to be triturated in a glaſs-mortar till it is reduced to as fine a

* Publiſhed in the 4th Vol. of the Tranſactions of the Berlin Society.

powder as is poſſible. This powder is to be mixed with twice as much charcoal-duſt, and being worked up with a little honey into a thick paſte, is to be ſubjected, in a covered crucible, to a ſtrong red heat for half an hour. When the veſſel is become cold, the hepatic maſs is to be taken out, and after it has been rubbed to a powder, marine acid diluted with ſix times its quantity of water is to be gradually poured upon it, till the efferveſence ceaſes. The liquor is afterwards to be heated, and the undiſſolved earth, which is undecompoſed ponderous ſpar, muſt be allowed to ſettle. The clear ſolution is then poured off, and the remaining powder edulcorated and dried. This powder muſt be again calcined with charcoal-duſt in the ſame manner as before, and the freſh hepar thus obtained muſt be diſſolved, like the former, in marine acid.

The muriatic ſolution thus prepared is to be put into a matraſs (the neck of which has been broken off) and to be ſubjected to digeſtion for ſeveral hours in a ſtrong heat, in order to expel all the ſtinking hepatic air.

This

This being done, the ſolution is filtered, and while it is yet as hot as poſſible, the diſſolved earth is precipitated from it by lixivium tartari, and the precipitate is edulcorated and dried.

§. III.

(1) This earth (the *terra ponderoſa aerata* of Bergmann) melts in a ſtrong fire, and after cooling is found to have acquired a bluiſh tinge, but not any tranſparency.

(2) Glaſs of borax diſſolves it for the moſt part, with effervеſcence. With microcoſmic ſalt the effect is the ſame.

A. The melted maſs (1) grows hot on the addition of acids.

B. Reduced to fine powder and boiled in water, it is in part diſſolved. There remains, however, a conſiderable portion, which is inſoluble in water, and which ſtill effervеſces with acids.

C. This watery ſolution of the earth in queſtion (which may be called *aqua terræ ponderoſæ*) is precipitated by fixed air, ſtrikes

a yellow colour with a ſolution of corroſive ſublimate of mercury, and diſſolves ſulphur.

D. If vitriolic acid is added to the aqua terræ ponderoſæ, a regenerated ponderous ſpar is immediately thrown down. The ſame effect takes place on adding a ſolution of ſelenite, vitriolated tartar, or Glauber's ſalt.

(3) Terra ponderoſa efferveſces with, but is not diſſolved by vitriolic acid.

(4) With the nitrous acid it gives a bitter ſolution which readily ſhoots into fine cryſtals, that are ſoluble in a large quantity of water, but are permanent in the air.

(5) With marine acid the effects are the ſame.

(6) Though it efferveſces with, yet it is ſcarcely at all diſſolved by the fluor, arſenical and phoſphoric acids. On the other hand, it not only efferveſces with, but is diſſolved by acid of tartar diluted with water. It is likewiſe ſoluble in diſtilled vinegar. This ſolution, ſubjected to evaporation, affords a gummy matter, which deliqueſces

liquefces in the air. The aërial acid, alfo, s capable of diffolving the ponderous earth.

(7) The nitrous and muriatic folutions of terra ponderofa are precipitated by fluorated ammoniac, arfenical ammoniac, tartarifed alkali, faccharine acid, and falt of woodforrel.

(8) Diftilled with fal ammoniac, the ponderous earth difengages the volatile alkali.

§. IV.

How far the earth of ponderous fpar differs from calcareous earth may be feen by what follows:

A. Ponderous earth is fufible in the fire.

B. The aqua terræ ponderofæ is decompofed by all neutral and middle falts which contain vitriolic acid.

C. With the nitrous and marine acids the ponderous earth forms cryftals that are permanent in the air. But,

D. With the acetous acid it yields a deliquefcent falt.

Hence it is evident that the heavy ſpar contains an earth of a peculiar nature.

III. OBSERVATIONS ON

CERUSSE or WHITE LEAD;

By Mr. SCHEELE*.

WHITE lead, diſtilled by itſelf, gives out an empyreumatic matter reſembling ſpirit of tartar, and fixed air. If vitriolic acid was previouſly added, there paſſes over into the receiver a little vinegar.

If ceruſſa alba be boiled in diſtilled water, and the water be afterwards filtrated, none of the lead will be found diſſolved in it.

* Publiſhed in Gottling's Almanack or Pocket Book for Chemiſts for the Year 1788.

Clear

Clear vinegar of lead, expoſed to the air, gradually depoſits a white powder. This white powder being collected and edulcorated is found to be ſimilar in every reſpect to white lead.

Ceruſſe is, in like manner, precipitated from vinegar of lead by the aërial acid.

Into a glaſs veſſel filled with vinegar, I introduced a plate of lead, and afterwards cloſed the mouth of the veſſel, ſo as to exclude the acceſs of the ſurrounding air. After ſome weeks, a little of the lead was diſſolved.

In like manner I introduced into a ſecond glaſs veſſel filled with vinegar that had been previouſly boiled, another plate of lead. After the ſpace of a whole year, I did not diſcover in this vinegar the leaſt veſtige of diſſolved lead.

I filled a glaſs veſſel with fixed air, and after pouring into it a little vinegar, I ſuſpended a plate of lead in the fixed air, and cloſed the mouth of the veſſel. But in this caſe, too, the lead was not corroded.

If pure lead be dipped in a ſolution of ſugar of lead, and hung up in the air, it will

be

be found covered over with ceruſſe, the next day.

If a plate of lead is kept in a ſolution of ſugar of lead, for the ſpace of a day, the liquor is converted into vinegar of lead; that is, the ſolution of ſugar of lead attracts pure ſuperfluous calx of lead. Accordingly, on the addition of a mucilage, for inſtance gum arabic, a coagulation takes place.

Hence it appears that not only vinegar, but alſo fixed air and pure air, are neceſſary for the production of white lead.

The acetous acid, which has no effect upon lead in its reguline ſtate, readily attacks this metal when previouſly dephlogiſticated by the air. The ſugar of lead thus produced attracts ſuperfluous calx of lead, as the lead becomes more and more dephlogiſticated. The ſuperfluous calx of lead is again precipitated by the aërial acid, and covers the plate of lead. In this way a ſmall quantity of ſugar of lead is capable of converting a large quantity of reguline lead into a calciform ſtate.

IV. EX-

IV. EXPERIMENTS

ON THE

ACID OF BENZOIN,

SHEWING ITS EFFECTS UPON

METALS AND THEIR CALCES;

By Mr. TROMMSDORF.

§. I.

ALTHOUGH the ſtudy of ſaline ſubſtances is a field in which our induſtrious chemiſts have laboured with great diligence, yet there are many parts of it which ſtill lie waſte and uncultivated. Our knowledge in particular reſpecting the combinations of many acids into neutral ſalts is ſtill very imperfect, and this is remarkably the caſe with the acids of amber and benzoin. Concerning this laſt, we only know from the experiments of Mr. Lichtenſtein in what manner it acts upon alkalis and earths; but

but with the order of its attraction to these substances, and more particularly with its effects upon metals, we are still unacquainted. This deficiency I have endeavoured to supply, and now communicate a part of my experiments, which I shall hereafter continue. In the farther prosecution of these inquiries, I shall examine the effects of this acid upon alkalis and earths, in order to determine its affinities, and ascertain more fully the properties of the neutral salts which it forms. Lastly, I shall make some experiments on the decomposition of the acid of benzoin by dephlogisticated marine acid, nitrous acid, manganese, oil of vitriol, &c.

§. II.

But before I describe the properties resulting from the combinations of this acid with metals, it is necessary to mention the method which I pursue in order to effect such combinations. It is as follows:

To one part of metal (granulated, in filings, or in leaf) or its calx, are added two parts

parts of acid of benzoin, and upon this mixture, which is made in a deep glaſs jar, as much hot diſtilled water is poured as is ſufficient for diſſolving the ſalt of benzoin. The mixture is then placed in a ſand-bath, where it is ſuffered to boil gently for ſeven or eight hours, during which the loſs by evaporation is repaired from time to time by the addition of freſh water. The liquor is then poured off, boiling-hot, into a filter of unſized paper, and the metal or calx which remains behind is edulcorated with diſtilled water.

This I found to be the only ſucceſsful mode of proceeding; for unleſs the mixture be kept boiling, the ſalt of benzoin ſhoots into cryſtals, in conſequence of being ſo little ſoluble in cold water.

§. III.

To avoid repetition I ſhall here mention how I prepared my metallic calces.

Calx of gold was precipitated by pure vegetable alkali, from a ſolution of gold in dephlogiſticated marine acid.

Calx

Calx of ſilver from a ſolution in nitrous acid, by mineral alkali.

Calx of platina from a ſolution in aqua regia, by mineral alkali. Some chemiſts * maintain that the mineral alkali occaſions no precipitation in this caſe; but I can affirm the contrary †. It is true, the precipitation takes place rather ſlowly; but this is the beſt mode of obtaining a pure calx of platina; for it is known that the volatile ‡ as well as the vegetable alkali, throws down platina not in the ſtate of a calx, but of a triple ſalt.

Calx of copper was precipitated from vitriol of copper by mineral alkali.

Calx of tin § and calx of lead were obtained in the uſual manner by calcination.

Calx of iron from vitriol of iron, by mineral alkali.

* Marggraf, Lewis, Wenzel.

† This has long ſince been proved by Bergmann.

‡ In the original we read "MINERAL as well as vegetable alkali;" but this muſt be a miſtake.

§ By an error of the preſs it is "*Zinkkalk*," inſtead of "*Zinnkalk*" in the original. Calx of zinc is mentioned in it's proper place afterwards.

Calk of quickſilver from nitrated quickſilver, by vegetable alkali.

Calx of biſmuth from nitrated biſmuth, by vegetable alkali.

Calx of zinc by calcination.

Calx of antimony by detonating pure regulus of antimony with nitre.

The calx of arſenic was common white arſenic, that was quite tranſparent.

Calx of manganeſe from pure vitriol of manganeſe, by vegetable alkali.

Calx of cobalt from vitriolated cobalt, by mineral alkali.

Calx of nickel from vitriolated nickel, by mineral alkali.

As I have not yet been able to procure any genuine molybdæna, tungſten, and uranium, experiments on theſe ſubſtances are ſtill wanting.

It is ſcarcely neceſſary to mention that all theſe calces were repeatedly lixiviated by boiling in diſtilled water, and afterwards thoroughly dried.

§. IV.

The acid of benzoin itſelf was procured according to profeſſor Gren's method, by

boiling

boiling the resin of benzoin with distilled water and mineral alkali, and afterwards decompounding the salt thus formed, by means of vitriolic acid. By repeated solution the acid of benzoin is brought to shoot into silver-white crystals.

§. V.

Gold. The acid of benzoin does not attack gold in its metallic state ; but when reduced to a calx, it dissolves it, though but sparingly. The solution shoots into small irregular crystals, which dissolve with difficulty in water, and scarcely at all in spirit of wine. They are permanent in the air, but in the fire their acid flies off, and, when the heat is sufficiently great, the gold remains behind in a metallic form.

§. VI.

Silver. Upon reguline silver the acid of benzoin produces no effect, and even very little of its calx is dissolved by it. An union is best effected by adding *benzoinated* * vegetable

* Vegetable alkali formed into a neutral salt with acid of benzoin.

alkali to a ſaturated ſolution of nitrated ſilver; a powdery precipitate is immediately let fall, and is found to be ſilver combined with the acid of benzoin (Benzoinated ſilver) It readily diſſolves in hot water, but ſeparates again by cooling. Very little of it is diſſolved by ſpirit of wine, even with the aſſiſtance of a boiling heat. It does not deliqueſce in the air, but is turned brown by the rays of the ſun. The acid flies off in the fire, and the reſiduum is ſilver revived.

§. VII.

Platina. In its metallic ſtate platina is not attacked by the acid of benzoin, but a ſmall portion of its calx is diſſolved by it. This ſolution ſhoots into yellowiſh ſtar-like cryſtals, which are permanent in the air, are difficultly ſoluble in water, and inſoluble in ſpirit of wine. The acid flies off in the fire, and the platina remains behind in the form of a yellow powder.

§. VIII.

Copper. Reguline copper is not at all affected by acid of benzoin; but its calx is

eaſily diſſolved by it. This ſolution is of a greeniſh colour, has a bitter taſte, and ſhoots into ſmall pointed cryſtals of a dark green colour. This ſalt is very difficultly ſoluble in water, and quite inſoluble in ſpirit of wine. Theſe cryſtals remain dry, but effloreſce a little in the air. They are decompoſed by mild and cauſtic alkalis, and by quicklime and cauſtic ponderous earth. Expoſed to the fire they part with the acid, leaving behind a pure calx of copper.

§. IX.

Tin. The acid of benzoin has no effect upon tin, either in its reguline or calcined ſtate. A ſolution of this metal, however, in aqua regia is in part decompoſed by benzoinated vegetable alkali, and the precipitate is benzoinated tin, which is ſoluble in boiling water only; none of it is taken up by ſpirit of wine. It is decompoſed in the fire.

§. X.

Lead. Reguline lead, after it has been boiled for a conſiderable length of time with

acid

acid of benzoin, becomes ſomewhat tarniſhed on its ſurface, and when diſtilled water that has been impregnated with hepatic air is added to the ſolution, a blackiſh colour is produced: But the quantity of lead diſſolved in this caſe is ſo trifling, that it can ſcarcely be detected by any other medium than the above-mentioned ſenſible teſt or re-agent.—Calx of lead, however, is very readily diſſolved by the acid of benzoin, and this ſolution, which has a ſweetiſh taſte, ſhoots into very white ſhining cryſtals, which lie very looſe one upon the other. They diſſolve readily both in diſtilled water and in ſpirit of wine. From theſe ſolutions the lead is precipitated black by hepar ſulphuris, and white, by mild alkalis and earths. Vitriolic acid throws down a vitriol of lead, and marine acid a plumbum corneum; conſequently the acid of benzoin has a weaker attraction to lead than thoſe acids. The above-mentioned cryſtals are permanent in the air, but when expoſed to the fire, they part with their acid, leaving behind a calx of lead.

§. XI.

Iron. The acid of benzoin attacks iron in its metallic ſtate; the quantity, however, which it diſſolves is extremely inconſiderable. It diſſolves the calx of iron better, though it cannot be completely ſaturated even with it. By gentle evaporation the ſolution yields yellowiſh, ſparry cryſtals, which have a ſweetiſh taſte. They remain dry, but effloreſce, in the air; and when they are diſſolved again in diſtilled water, a conſiderable quantity of ochre or calx of iron is depoſited in the form of yellow flakes. Spirit of wine, alſo, readily diſſolves this ſalt (benzoinated iron) and from this ſolution, too, as well as the former, a calx of iron ſeparates.

Infuſion of galls communicates a black, and lixivium ſanguinis a fine blue colour to theſe ſolutions; and alkalis, whether mild or cauſtic, precipitate a calx of iron from them: they are likewiſe decompoſed by quicklime and cauſtic ponderous earth. In the fire the acid volatilizes a portion of the iron;

iron; the remainder appears in the ſtate of a calx.

§. XII.

Quickſilver. In its metallic ſtate quickſilver ſuffers no loſs of weight when boiled with acid of benzoin, nor is any precipitation produced in the liquor, after digeſtion therewith, by vitriolic or marine acid. Calx of quickſilver, however, is pretty readily diſſolved by this acid, but not to the point of ſaturation. By gentle evaporation no cryſtals are obtained, but merely a white powdery maſs, which is very brilliant, and ſuffers no alteration by expoſure to the air. It is ſomewhat ſoluble in ſpirit of wine, but ſcarcely any of it is taken up by water. Mild alkalis precipitate a white calx of quickſilver, and lime-water a pale yellow calx, from theſe ſolutions. This ſalt is likewiſe decompoſed by the vitriolic and marine acids, and liver of ſulphur produces a dark coloured precipitate. In the open fire this ſalt is completely volatilized and diſſipated; in cloſe veſſels it ſublimes on the

application of a gentle heat; but if the heat be too great, the acid ſeparates, and the quickſilver appears in a metallic form.

Might not benzoinated quickſilver be employed with advantage as a medicine?

The acid may be united with quickſilver to the point of ſaturation, by adding benzoinated vegetable alkali diſſolved in diſtilled water, to a ſaturated ſolution of quickſilver in nitrous acid. The precipitate thus obtained is to be collected, and edulcorated with cold diſtilled water.

§. XIII.

Biſmuth. Reguline biſmuth is not attacked by acid of benzoin; but its calx is readily acted upon, and with effervesſcence. The ſolution yields a light maſs, which conſiſts of white cryſtalline ſpiculæ. This ſalt is not deliqueſcent, is ſomewhat ſoluble in ſpirit of wine, and is readily diſſolved by boiling water, from which, however, a portion of calx afterwards ſeparates. It is decompounded by vitriolic and marine acid, and parts with its acid in the fire.

§. XIV.

§. XIV.

Zinc. The acid of benzoin has but little action upon zinc in its metallic ſtate; but it readily diſſolves calcined zinc, not however ſo as to be completely ſaturated therewith. The ſolution is clear as water, has a ſweet ſtyptic taſte, and yields arboreſcent cryſtals, which effloreſce by expoſure to the air, and diſſolve readily both in ſpirit of wine and in water. Alkalis, quicklime and cauſtic ponderous earth decompoſe this ſalt, and its acid flies off in the fire.

§. XV.

Antimony. Regulus of antimony is not diſſolved by acid of benzoin, but its calx is. The ſolution does not afford any true cryſtals, but only a white flaky maſs, which diſſolves readily both in water and in ſpirit of wine. From theſe ſolutions the calx of antimony is precipitated of a white colour by alkalis, whether aerated or cauſtic, and alſo by quicklime and cauſtic ponderous earth. Hepar ſulphuris precipitates a pale

yellow ſulphur of antimony. This ſeems to be owing to the ſuperfluous acid of the benzoinated antimony, which precipitates along with the pure ſulphur, and which, mixing with the ſulphur of antimony that afterwards falls down, renders this laſt of a lighter colour. Benzoinated antimony remains dry in the air, and is decompoſed in the fire. On dropping pure marine acid into a ſolution of this ſalt, the pure acid of benzoin ſeparates nearly in a cryſtallized ſtate; a proof that benzoinated antimony is more ſoluble in water than pure acid of benzoin, and that antimony has a ſtronger affinity to the marine acid than to the acid of benzoin.

§. XVI.

Manganeſe. Upon regulus of manganeſe the acid of benzoin has no effect; its black calx is in ſome meaſure diſſolved by it, but it acts beſt upon the white (phlogiſticated) calx, which is precipitated from acids. This ſolution is of a bright (or reddiſh) brown colour, and has a very ſweet taſte.

By

By gentle evaporation it yields ſmall, ſcaly cryſtals, which diſſolve readily in water, but difficultly in ſpirit of wine. Alkalis, quicklime and cauſtic ponderous earth precipitate the calx of manganeſe from theſe ſolutions. The cryſtals remain unaltered in the air, and in the fire a brown calx is left behind.

§. XVII.

Nickel. This ſemi-metal in its reguline ſtate is not attacked by acid of benzoin, but in the ſtate of a calx it is ſparingly diſſolved by it. The ſolution is of a pale green colour; it yields no cryſtals, but only a flaky maſs, which effloreſces in the air. This ſalt diſſolves readily in diſtilled water, and in ſome meaſure too in ſpirit of wine. Tincture of litmus is reddened by this ſolution, which is decompoſed by alkalis, quicklime, and cauſtic ponderous earth. The acid flies off in the fire.

§. XVIII.

Arſenic. Regulus of arſenic is indeed attacked, though very ſlowly, by acid of benzoin;

benzoin; the white calx, however, is readily diſſolved by it, and the ſolution yields a ſalt which cryſtallizes into the form of plumes, the feathers of which are very long and finely pointed. Of all the benzoinated ſalts, this exhibits the moſt remarkable cryſtallization. This ſalt has a manifeſtly acid and ſomewhat pungent taſte. Expoſed to the air, it loſes its water of cryſtallization, and effloreſces a little. It diſſolves readily in hot diſtilled water, but cryſtallizes again on cooling. Neither mild nor cauſtic alkalis decompoſe its ſolution; but liver of ſulphur throws down a precipitate, which is yellow arſenic. On the application of a moderate heat, the ſalt is ſublimed in a flocculent or downy form, but in a ſtrong fire the combination is deſtroyed.

§. XIX.

Cobalt. Regulus of cobalt is not attacked by acid of benzoin, and even its calx is but ſparingly diſſolved by it. The ſolution yields cryſtals, which are nearly of the ſame figure as thoſe of the pure acid of benzoin.

From

From their ſolution, however, in diſtilled water a little metallic calx is precipitated by the vegetable alkali.

§. XX.

The middle ſalts formed by the union of pure acid of benzoin with metals (§. 5. to §. 19.) are eſſentially different from all other ſaline compounds; conſequently the acid of benzoin muſt be conſidered as a peculiar acid. We may conjecture from analogy, that its baſis is the general vegetable acid; but to prove this, many deciſive experiments are wanting.

As the acid of benzoin detonates with nitre, it neceſſarily contains the inflammable principle; and it ſeems to me to be owing to the preſence of this principle, that it acts ſo little upon metals in their reguline ſtate. At leaſt this is known to be the caſe with other acids, for inſtance, marine acid, which is not capable of diſſolving gold 'till it has been deprived of its phlogiſton.

§. XXI.

If a plate of copper be ſuſpended in a ſolution of benzoinated ſilver, this laſt is precipitated in its metallic ſtate, and the copper is diſſolved. It ſeems at firſt extraordinary, that the acid of benzoin, which does not act upon reguline copper (by itſelf) ſhould in this inſtance diſſolve it, and let go the ſilver; but on conſidering the matter more fully, it will appear probable, that the calx of ſilver is the principal agent in this caſe, in conſequence of having a ſtronger affinity to phlogiſton than the calx of copper has. Hence, what could not otherwiſe be effected, is thus brought about by means of a double elective attraction. We may therefore preſume, that the ſame order in which the metals are precipitated by one another from their ſolutions in other acids, obtains here.

I ſhould have been glad to have aſcertained the order of affinities of the metallic calces to the acid of benzoin. This inquiry, however, is attended with numerous difficulties; and

and as I have not yet been able to obtain any reſults that are ſufficiently accurate, I muſt defer this matter to ſome other time.

* *

V. PROCESS FOR OBTAINING KUNKEL's PHOSPHORUS FROM URINE,

IN A SHORTER TIME AND AT LESS EXPENCE THAN BY SCHEELE's AND GAHN's METHOD WITH BONES;

By Mr. GIOBERT of the Turin Academy*.

IT was long ſince remarked by the celebrated Marggraf, that when either calx of lead, or plumbum corneum, was added to urine, the quantity of phoſphorus which it yielded was conſiderably increaſed. He

* Extracted from the Annales de Chimie for January 1792.

was

was unable, however, to account for this fact; for it was not then known that urine contains two different phoſphoric ſalts, one that has the volatile, and another the mineral alkali, for its baſis: nor was it known that the laſt mentioned compound, phoſphorated ſoda, was unſuſceptible of decompoſition by treatment with charcoal*; and conſequently, that the quantity of phoſphorus obtained in the diſtillation of the urine, could only be in proportion to the quantity of phoſporic ammoniac which the urine contained. Moreover, as the nature of the phoſphoric acid itſelf was not well underſtood by the chemiſts of thoſe days, they were of courſe unacquainted with the laws of affinity which it follows in its different combinations. They could not ſuppoſe that plumbum corneum was capable of ſeparating the mineral alkali from its union with

* Mr. Wenzel makes charcoal capable of decompounding phoſphorated ſoda, and of converting, at the ſame time, the phoſphoric acid into phoſphorus. But Mr. Giobert remarks, that this fact has been called in queſtion by Dr. Crell, and that he does not know of any chemiſt, beſides Mr. Wenzel, who has ever obtained ſuch a reſult.

with the phoſphoric acid; nor had they any notion that phoſphorated lead could be decompoſed by charcoal, which reduces the calx of lead, and converts the phoſphoric acid into phoſphorus. This fact was not proved till a long time after by ſome French chemiſts in their treatment of phoſphorated lead with charcoal powder, and more eſpecially by Mr. Delametherie, in his experiments on the green lead ore from Hoffsguard, in Briſgaw. After this it was eaſy to ſee what took place in Mr. Marggraf's experiments, and the advantage derived from the addition of the plumbum corneum to the urine is now ſufficiently obvious. The marine acid of the plumbum corneum ſeizes the baſis of the phoſphoric ammoniac and phoſphorated ſoda, whilſt the diſengaged phoſphoric acid joins itſelf to the calx of lead deſerted by the marine acid. The new compounds formed by means of this double affinity are ſal ammoniac and common ſalt, and phoſphorated lead. This laſt product is decompoſed by charcoal, and the phoſphoric acid is thereby converted into phoſ-

phorus

phorus. Hence the increaſed quantity of phoſphorus in Mr. Marggraf's proceſs.

It only remained to try whether this combination ſucceeds equally well in the humid way, or with urine in the ſtate in which it is diſcharged from the body, and before it has undergone a tedious and offenſive evaporation. For if the decompoſition of the phoſphoric ſalts of urine is effected by means of plumbum corneum, or any ſalt having calx of lead for its baſis, the phoſphorated lead, being inſoluble in water, ſhould fall to the bottom, and the precipitate in this caſe, ſhould preſent all the phoſphoric acid contained in the urine, and preſent it, too, in a ſtate very fit for being converted into phoſphorus. It is upon theſe principles that the new method here propoſed is founded.

In ſome experiments with phoſphoric acid obtained from bones, Mr. Giobert found that this acid ſeparates the calx of lead from its ſolution in the acetous, and even the nitrous acid; and that as the compound of lead and phoſphoric acid is inſoluble

ſoluble in water, a precipitation takes place.

Expecting that the decompoſition of ſalts that have calx of lead for their baſis, would be ſooner and more completely effected by means of double attractions; Mr. Giobert tried to decompoſe the phoſphoric ſalts of urine by a ſolution of lead in the acetous acid. The event anſwered his expectations. By pouring vitriolic acid upon the precipitate obtained in this manner from urine, diluting the mixture with water, filtrating the liquor and ſaturating it with volatile alkali, he procured cryſtals which had all the properties of phoſphoric ammoniac. The reſiduum was nothing but vitriolated lead. In this caſe, therefore, the phoſphoric acid left the mineral and volatile alkalis to join itſelf to the calx of lead deſerted by the acetous acid.

By further experiments it was found, that, on decompounding the phoſphoric ſalts of urine by a ſolution of lead in the acetous acid, independently of the phoſphorated lead which is depoſited, there is alſo preci-

pitated a portion of calx uncombined with phoſphoric acid. This inconvenience (which is owing to the property that this ſolution has of being decompoſed by water) may be eaſily remedied, by employing a ſolution of lead with a ſmall exceſs of the acid. The ſame reſult is obtained with a ſolution of lead in nitrous acid.

If, inſtead of nitrated or acetated lead, a ſolution of zinc in vitriolic acid be poured into urine, the phoſphoric ſalts of the urine are in like manner decompoſed, and phoſphorated zinc is precipitated. This is ſuſceptible of decompoſition, like phoſphorated lead, by treatment with charcoal, which, at the ſame time that it revivifies the zinc, converts the phoſphoric acid into phoſphorus.

For this purpoſe, however, the employment of vitriolated zinc is not found ſo proper as might at firſt be ſuppoſed; for in the diſtillation of the phoſphorated zinc obtained from the decompoſition of urine by vitriolated zinc, ſome of the revived metal is ſublimed into the neck of the retort, part of

of the phoſphorus that is formed combines with the zinc, and very little phoſphorus paſſes into the receiver. Mr. de Morveau has made mention of this fact in his remarks upon a proceſs of the celebrated Dr. Crell's. The ſame inconvenience happens if nitrated quickſilver is employed. This ſalt decompoſes the phoſphoric ſalts of urine very well, and the precipitate thus obtained, viz. phoſphorated quickſilver, is decompoſable by charcoal; but in this caſe a portion of the mercury conſtantly unites with the phoſphorus, and ſome of this paſſes over into the receiver, thereby rendering the phoſphorus impure.

Many other metallic ſolutions were found to decompoſe the phoſphorated ſalts of urine in the ſame manner, and to yield phoſphorus by diſtillation with charcoal; but as none of them anſwered ſo well as nitrated lead, Mr. Giobert has thought it unneceſſary to enter into a detail of thoſe experiments.

By means of nitrated lead, phoſphorus may be procured from urine in the courſe

of a day, or even a few hours. It is by no means neceſſary (as was formerly ſuppoſed) that the urine be putrid; on the contrary, freſh urine ſeems to be preferable. It appears to be a matter of indifference whether the urine employed for this purpoſe be taken from healthy or ſick perſons; and the ſtale of horſes was found to anſwer nearly as well as human urine.

(PROCESS.)

A ſolution of lead in nitrous acid is poured little by little into the urine, till there ceaſes to be any conſiderable precipitation. A large quantity of water is then added for the attenuation or dilution of the extractive matter of the urine. The mixture is afterwards filtered through a linen ſtrainer, and the phoſphorated lead which is left upon the filter is made into a paſte with charcoal powder. This paſte is dried, put into a retort, and ſubjected to diſtillation. There comes over, firſt an oily volatile alkali in a fluid form; afterwards a little empyreumatic oil, both which proceed from the urine, from which it is difficult

cult wholly to free the phoſphorated lead. As ſoon as the oil ceaſes to come over the receiver is changed, the heat is conſiderably increaſed, and a receiver with water (in the uſual manner) is fitted to the retort. The phoſphorus makes its appearance ſometimes in the courſe of half an hour; and an operation capable of yielding twelve or fourteen ounces of phoſphorus may be eaſily finiſhed in eight hours time. Mr. Giobert thinks that the violent degree of heat which is applied in the diſtillation of phoſphorus prepared from bones, according to the ordinary method, is not here abſolutely neceſſary.

Lead diſſolved in vinegar may be employed inſtead of nitrated lead; but in this caſe there is a greater quantity of the metal in the ſtate of a calx, uncombined with phoſphoric acid. In thoſe places, however, where ſugar of lead is manufactured in the great, it may be preferable, from its cheapneſs, to nitrated lead. When acetated lead is employed, it will be proper to ſubject the urine, after the addition of this ſalt, to a di-

gesting heat for some hours, and even to add a little vinegar.

The quantity of phosphorus obtainable from a given weight of phosphorated lead cannot be precisely determined. The nature of the urine may occasion some difference, and much depends on the care which is shown in the edulcoration or washing. When the phosphorated lead is thoroughly washed, and the calx* is completely saturated with phosphoric acid, Mr. Giobert thinks he may assert from his experiments, that 100 parts of phosphorated lead yield from 14 to 18 of phosphorus.

The calx of lead is found reduced at the bottom of the retort, and this compensates a part of the cost of the materials employed. The phosphorus thus obtained is contaminated with some oily matter, which it dissolves in passing through the neck of the retort; but it is easily freed from this impurity by Mr. Wolfe's method.

* By an error of the press it is "*acide*" instead of "*oxide*" in the original.

By the ſame proceſs may be obtained in a very expeditious manner phoſphoric ammoniac for aſſays with the blow-pipe. A ſolution of vitriolic ammoniac is added to the phoſphorated lead precipitated from urine, and the mixture is digeſted for 12 hours. The liquor is then filtered; and by gentle evaporation it ſhoots into cryſtals of phoſphoric ammoniac.

In like manner if a ſolution of Glauber's ſalt is poured upon phoſphorated lead, the vitriolic acid ſeizes the calx of lead, and precipitates it, and the phoſphoric acid joins itſelf to the mineral alkali. The filtrated liquor yields by evaporation cryſtals of phoſphorated ſoda.

VI. EXPERIMENTS

ON

MOLYBDÆNA.

WITH A

VIEW TO ITS REDUCTION;

By Mr. HIELM.

(*Continued.*)

§. VII.

IN the manner already described Mr. Hielm provided himself with a quantity of earth of molybdæna free from sulphur and its acid, and consequently in a state suited to experiments for its reduction.

Of this earth a small quantity, previously levigated and moistened with a little linseed oil, so as to make its particles stick together, was put into a crucible, which had

had been lined with charcoal-powder, in the uſual manner. The crucible was then filled half way up with charcoal-duſt, a cover was luted to it, and it was expoſed for half an hour to the heat of a forge-furnace which anſwered very well for making common aſſays of iron. On breaking the crucible, the molybdæna was found caked or agglutinated into a pretty hard lump, and to be rather of a darker colour than before; but no marks of perfect fuſion, nor any metallic globules could be diſcovered in it, on the application of a magnifying glaſs.

This lump of molybdæna, without being pulveriſed, was again expoſed (under the ſame circumſtances as before) to a greater degree of heat, and for a greater length of time; after which, it appeared ſomewhat of a darker colour, probably in conſequence of the abſorption of more phlogiſton. This operation was repeated four times, with a conſtantly increaſed heat, continued for an hour, and more; but it thereby ſuffered little alteration. There were, indeed,

on

on the outſide of the lump, ſome globules of a browniſh ſcoria, on which were diſcoverable particles or dots of a metallic appearance; but ſuch an appearance could not be conſidered as a proof of a metallic reduction.

§. VIII.

It was now concluded that the earth of molybdæna had, by this treatment in the fire, abſorbed phlogiſton enough for its metalliſation, and that nothing more was wanting for bringing it into fuſion, but the addition of a proper flux. Accordingly, the above-mentioned agglutinated maſs was reduced to powder. Under the hammer it was found to be pretty hard, but brittle. Internally it was of a greyiſh colour, with ſhining ſpots, finely grained, and of a metallic appearance. Some of this pulveriſed earth of molybdæna was put into a ſmall crucible, the bottom of which had been previouſly beſmeared with linſeed oil, and afterwards duſted over with charcoal powder. A little charcoal powder was laid over the

the molybdæna itſelf, and laſtly over this was put as much glaſs of borax as was thought ſufficient for covering the expected metallic button. A cover was luted to this crucible which was placed in a larger one, furniſhed with a looſe cover; and the whole was then expoſed to the ſtrongeſt heat of a forge for half an hour. The glaſs of borax was thereby changed to a dark green colour, but no regulus was found at the bottom of the crucible. On the ſurface of the ſcoria, there appeared ſome ſmall metallic grains, remarkably brilliant, and of a white colour. When ſeparated, they were found to be attracted by the magnet, and were eaſily ſcratched and cut with a penknife. Internally they were of a bluiſh grey colour, and of a ſcaly or laminated texture.

Nothing ſatisfactory was derived from this experiment. It only ſerved to ſhew, that the flux here employed does not contribute to the reduction of the molybdænic calx. The ſmall trace of iron (from which it is difficult wholly to free the earth of molybdæna) tends, in Mr. Hielm's opinion, rather

ther to promote than impede its metallisation.

§. IX.

In consequence of the violent and long continued heat to which they are exposed, the crucibles in these experiments sometimes crack and break asunder, in which case the calx of molybdæna is lost, and the labour thrown away. On this account it is proper to subject to these trials only very minute quantities, and in the smallest possible crucibles; but as it is difficult to manage such small crucibles by themselves, when a strong blast is applied, it was found best to make several assays at once. In this way, not only time and labour were saved, but an opportunity was also given of seeing how far the molybdæna might be more or less disposed to melt, according as it should be situated more or less above or below the stream of air from the bellows.

Accordingly

Accordingly ſeveral crucibles were lined with charcoal powder, and into the largeſt of them was put as much as a quintal or 100 aſs. of earth of molybdæna; this quantity, however, was generally divided into four parts. A layer of charcoal powder was ſtrewed over the molybdæna, and over this was put a piece of an old broken crucible, which was to ſerve as a baſis or ſtand to another ſmaller crucible. This ſmaller crucible (furniſhed with charcoal like the former, but containing only 16 aſs. of molybdænic earth) was fixed in the larger one, ſo that its angles or corners touched the ſides of the outer one. In this ſmall crucible another ſtill ſmaller, with a proportionably leſs quantity of molybdæna may be frequently placed; and in the angles or corners of the largeſt or outermoſt crucible, there is ſtill room enough for three of the very ſmalleſt crucibles*.

* Mr. Hielm took advantage of this circumſtance for trying, at the ſame time, the effects of the calx of molybdæna (and alſo of the reduced molybdæna itſelf) upon metals and their calces. In theſe experiments he found (as will be afterwards ſeen) that the calx of molybdæna mixed, in certain proportions, with other metals, does

In this apparatus the calx of molybdæna was expoſed for the ſpace of an hour to a degree of heat that had been found ſufficient for the fuſion of eight aſſays of iron at once. After the operation was over, the molybdæna in all the crucibles weighed 25 per cent. leſs than before; but in the buſineſs of fuſion, no advancement was made.

§. X.

From theſe failures Mr. Hielm concluded, that either charcoal was not ſo proper for furniſhing the calx of molybdæna with phlogiſton, as ſome other inflammable ſubſtances; or that this calx, when once phlogiſticated does not require a further addition of the inflammable principle, but that it muſt rather part with a little of its phlogiſton before it can be brought into fuſion; in other words, that the acceſs of air is neceſſary to its reduction.

Agreeably to theſe notions, bullock's blood was tried inſtead of charcoal; but the

does not impede their fuſion, and that even platina (in the ſtate in which it comes to us) treated with a ſmall quantity of molybdænic calx, melts into a perfect button, whoſe ſpecific gravity is 20°.

result

refult was the fame. Tallow, however, feemed to anfwer much better. A portion of the calx of molybdæna moiftened therewith, and afterwards expofed to the blaft without any further addition, was found to be more fcorified on its furface. After the lump or agglutinated mafs (previoufly dipped each time in melted tallow) had been repeatedly fubjected to a melting heat, it was at laft found covered over with a white vitreous fcoria, that looked like drops upon the thin flag. The lump of molybdæna under this flag appeared, efpecially when viewed with a magnifying glafs, very much like a metallic button. This button, on the furface next the fcoria, was of a clearer white than in the middle, where it was rather greyer. When this flattifh mafs was thrown upon an iron plate, it rung like a bit of any hard metal. Under the hammer it was pretty hard, but brittle. It was not fenfibly attracted by the magnet.

No nearer approaches to reduction were made by fubjecting a lump of molybdænic earth, that had been previoufly phlogifticated

ticated, to a ſtrong melting heat, in a covered crucible without any addition; but when, by means of an opening in the luting of the crucible, the air got acceſs to the molybdæna during its expoſure to the fire, it appeared to be ſomewhat more inclined to fuſion. Hence Mr. Hielm is induced to think that if the acceſs of air is not neceſſary, it is at leaſt not prejudicial to the fuſion of the molybdæna, provided it have been ſufficiently phlogiſticated beforehand, and more eſpecially if its ſurface be incruſted with a ſcoria.

§. XI.

Mr. Hielm now thought of another expedient. This was, to increaſe the degree of heat by means of manganeſe. With this view he filled ſeveral ſmall crucibles with crude *(rohem)* manganeſe, and luted covers upon them, ſo, however, as to leave a ſmall aperture in each for the purpoſe of giving vent to the pure or dephlogiſticated air that ſhould be extricated. Two of theſe crucibles were placed under the *form*

or

or place where the nozzle of the bellows enters, and four others at the bottom of the fire-place, round the pedeſtal that ſupported the aſſay or crucible containing the molybdæna. When the fuel was duly lighted, the blaſt was applied at firſt gently, and only with a ſmall weight upon the bellows, which, however, were afterwards worked with the uſual load. The fire was exceedingly briſk, and the fuel was rapidly conſumed *; but after half an hour's expoſure to this degree of heat, the calx of molybdæna was not found to be brought any nearer to fuſion than before.

Two crucibles, filled as before with manganeſe, were placed under the form. The bellows were ſet to work, and in five minutes a third crucible filled with manganeſe was placed among the fuel, on the other ſide of the aſſay †. Freſh crucibles

* On this occaſion Mr. Hielm was led to examine how much pure air a given quantity of manganeſe is capable of yielding, how long a time it takes for the diſcharge of all its pure air, &c. &c. Theſe experiments are related in a ſeparate paper.

† The pedeſtal which ſupported the aſſay was an inverted crucible, under which a ſmaller crucible filled with manganeſe, had been previouſly placed.

containing manganese were introduced, in this manner, every five minutes. In three quarters of an hour the fire-place began to be full of crucibles, which amounted to 13 in number, and contained in the whole two pounds of manganese. The fire was exceedingly strong, and the flame round the assay-crucible was dazzling white. The blast was continued a quarter of an hour after the introduction of the last crucible. At the end of the operation, it was found, contrary to expectation, that the calx of molybdæna had not yet been brought to a state of perfect fusion.

As it appeared, however, that the reduction was in some measure promoted by this treatment, Mr. Hielm was encouraged to persist in his attempt to overcome this refractory substance.

§. XII.

Sixteen ass. of calx of molybdæna were mixed with two ass. of plumbago or black lead. The inside of the crucible was rubbed with plumbago; the molybdæna and plumbago,

plumbago, after being worked together into a paſte with a little water, were put into the crucible, and over the whole was ſtrewed a thin layer of plumbago. The mixture melted into a ſpungy ſcoria, of a pale yellow colour, in which (to the naked eye at leaſt) there was not perceivable the ſmalleſt veſtige of the plumbago that had been added. From this circumſtance, as well as from the pale colour of the melted maſs, Mr. Hielm concluded that the quantity of black lead here employed was too ſmall. But before he tried the effects produced by melting theſe two ſubſtances together in other varied proportions, he reſolved to examine ſtill further the reſults obtained with the above-mentioned quantities. Of theſe experiments an account will be given in our next number.

VII. EXPERIMENTS WITH

SOLUTIONS OF MAGNESIA,

SHEWING THE

EFFECTS THEREOF IN DYING;

By Mr. VOGLER.

§. I.

PURE magnesia, prepared from Epsom salt in the best manner, and thoroughly edulcorated, was gradually thrown into oil of vitriol, spirit of nitre, and spirit of salt. Each of these acids dissolved it, in the cold, quickly and with effervescence, and the solutions were clear, till the moment of saturation, when a portion of the earth separated, of which part remained suspended on the surface of the liquor, and part settled to the bottom: and none of this, after the solutions were completely saturated,

rated, appeared to be rediſſolved either on the addition of more acid, or of water, or on the application of heat.

§. II.

Each ſolution of magneſia was diluted with three times its own quantity of pure water. Woollen, ſilken, linen and cotton ſtuffs, that had been previouſly ſcoured well, cleanſed and dried, were put into theſe ſolutions, and left to ſoak therein, without heat, for 10 or 12 hours; after which they were taken out, waſhed in three ſeparate quantities of clean, cold water, wrung out and dried.

§. III.

I now ſet over the fire three deep earthen pots, each of which contained two drams of madder and a full pint (between 12 and 14 ounces) of clean, ſoft water. After they had boiled for a few minutes, I put into one of the decoctions the bits of cloth or ſamples that had been prepared and ſteeped in the

vitriolic ſolution of magneſia; into another thoſe which had been ſoaked in the nitrous ſolution of magneſia; and into the third, thoſe which had been prepared in the muriatic ſolution of magneſia. After they had boiled therein for about ſeven or eight minutes, during which they were frequently turned over and preſſed down again with a ſtick, they were taken out of the decoctions, wrung out in two or three freſh quantities of clean cold water, and dried in the ſhade. The wool and ſilk were dyed throughout of a full bright brown colour, and the linen and cotton of a pale red.

§. IV.

Similar experiments were at the ſame time made with ſolutions of calcareous earth in the vitriolic, nitrous, and marine acids; and the colours thereby communicated to the ſtuffs that were afterwards boiled with madder, were found to differ little or not at all from thoſe obtained with the ſolutions of magneſia.

§. V.

§. V.

In like manner, alum and the different ſolutions of its earth, gave (as I found by repeated trials) in dying with the madder decoction, to wool and ſilk a light brown, and to linen and cotton, a red colour. Theſe colours, however, were always much brighter, finer and fuller than thoſe produced with the ſolutions of magneſia and calcareous earth.

From theſe and other comparative trials which I have made with ſolutions of the aluminous, magneſian and calcareous earths, in the dying of ſtuffs with logwood, braſilwood *(Fernambuck)* &c. it follows, that the magneſian and calcareous ſolutions are of little uſe in dying; that alum and its ſolutions produce the ſame, nay even finer colours, and conſequently may at all times ſupply their place.

It might alſo have been worth while to have tried, in the ſame manner, the effects produced by ſolutions of ponderous earth, and likewiſe of manganeſe (freed from its

heterogeneous parts) and its calx, in the dying of ſtuffs with the above-mentioned dyes; but for want of a proper quantity of heavy ſpar, and of time and opportunity to ſeparate the earth from the ſpar, and alſo to prepare pure manganeſe and its calx, I have not yet been able to undertake ſuch experiments.

VIII. ON THE PREPARATION OF

GLAUBER's SALT,

FROM

MARTIAL VITRIOL AND COMMON SALT;

By Mr. TUCHTEN.

IN the third number of the Chemical Annals for 1789, I find that Dr. Hahneman has not been able to prepare Glauber's ſalt from vitriol of iron and common ſalt, though

'though according to Mr. Ballen it is what may be eaſily done. If on repeating experiments, we ſhould not, in conſequence of ſome variations in the mode of performing them, always obtain the ſame reſults, we are not immediately to reject ſuch experiments as impoſitions. Without ever having read Mr. Ballen's account of his method, I have very often prepared (and I muſt ſay in an eaſy and cheap manner) Glauber's ſalt from green vitriol and ſea-ſalt; and if the directions hereafter mentioned be duly attended to, I am perſuaded that it will be found that what Mr. Ballen has aſſerted is not a mere ſpeculation, but a matter of fact.

When experiments are publiſhed, the circumſtances reſpecting the mode of conducting them cannot be too minutely deſcribed. I have not (as I ſaid before) read Mr. Ballen's account of his proceſs, but I dare ſay he has neglected to mention ſome of the neceſſary particulars; and hence Dr. Hahneman, failing in his trials, has been eaſily led to make oppoſite concluſions.

The

The ſucceſs of the operation entirely depends on the due regulation of the heat; for, if the mixture of martial vitriol and common ſalt be expoſed to too violent and long continued a degree of heat, the vitriolic acid is volatilized, and the conſequences are ſuch as Dr. Hahneman has ſtated: but, if 16 ounces of vitriol of iron, and 20 ounces of common ſalt be mixed together, and expoſed in a crucible to a gentle melting heat, only till the water of cryſtallization is evaporated, and till the maſs begins, at the ſides of the crucible, to enter into actual fuſion (for the firſt liquefaction was merely a ſolution in the water of cryſtallization, effected by the heat) and the crucible be now taken out of the fire, its contents lixiviated in diſtilled water made boiling hot, the lixivium filtrated, evaporated, and ſet by to cryſtallize;—the fineſt cryſtals of Glauber's ſalt will be obtained in the courſe of 24 hours. Theſe cryſtals, it is true, are impregnated with iron, from which, however, they may be purified by another ſolution and cryſtallization. To be certain of having a

thoroughly

thoroughly pure Glauber's ſalt, I add, in the ſecond ſolution, a ſmall quantity of mineral alkali, which ſerves to ſeparate the ſlight portion of ferruginous earth. In this manner, 16 ounces of green vitriol, and 20 ounces of common ſalt yield 12 ounces of pure Glauber's ſalt.

From 16 ounces of alum and 30 ounces of common ſalt treated in the ſame way, I have likewiſe obtained a pure Glauber's ſalt*.

* Another of Dr. Crell's correſpondents, Profeſſor Lieblein, of Fulda, bears teſtimony to Mr. Ballen's veracity in this inſtance, and aſſerts that he has twice prepared, in the preſence of thoſe who attend his academical lectures, the fineſt Glauber's ſalt from vitriol of iron, and common ſalt.

XI. LIQUOR

IX. LIQUOR FOR DETECTING IN WINES THE PRESENCE OF METALS, THAT ARE INJURIOUS TO THE HEALTH;

By Dr. HAHNEMAN*.

THE property which liver of ſulphur and hepatic air poſſeſs of precipitating lead of a black colour, has long been known; and advantage has been taken of this circumſtance for determining the purity of wines, in the preparation of the *liquor probatorius Wurtembergicus.*

But in the examination of wines ſuſpected to be adulterated, this teſt is not to be depended upon, ſince it precipitates iron of the ſame colour as it does the poiſonous metal, lead. Hence many reſpectable wine merchants have been undeſervedly thought

* Journal de Phyſique, for October, 1791.

to

to be guilty of the practice of adulteration, and been accordingly ruined.

There was ſtill wanting therefore a teſt or re-agent that ſhould diſcover in wines the preſence of ſuch metals only as are injurious to the health. Such a teſt is the following liquor, which precipitates lead and copper black, arſenic of an orange colour, &c. Without, however, precipitating iron, which, being harmleſs, or rather in many caſes ſalutary to the human conſtitution, gets, unheeded into wines on various occaſions.

(Preparation of the probatory liquor)

Mix together equal parts of oyſter-ſhells and brimſtone, both finely pulverized, and put the mixture into a covered crucible, which is to be placed in a wind-furnace. After it is thoroughly heated, the fire muſt be ſuddenly urged 'till the crucible becomes of a white heat, and continues ſo for a quarter of an hour. The maſs when cold, is to be reduced to powder, and kept in a bottle well ſtopped.

In

In order to prepare the liquor, 120 grains of this powder, and 180 grains of cream of tartar are put into a very ſtrong bottle, which is filled up with common water, that had been previouſly boiled for an hour, and afterwards ſuffered to cool. The bottle is then immediately corked, and ſhaken from time to time. After it has ſtood at reſt for a few hours, the clear liquor is decanted into ſmall phials capable of holding an ounce, 20 drops of ſpirit of ſea-ſalt being previouſly dropped into each of them. The mouths of the phials are then well cloſed with ſtopples made of wax and turpentine.

One part of this liquor mixed with three parts of the wine which is to be examined, will diſcover by a very manifeſt black precipitate, the ſlighteſt impregnation of lead, copper, &c. but will have no effect upon any iron that may be therein contained.

After this precipitate has ſettled to the bottom, we may find out whether there is alſo any iron, by ſaturating the decanted liquor with a little ſalt of tartar; if there be

be any iron, the liquor will immediately turn black.

Pure and unadulterated wines remain clear after the addition of this liquor.

X. NEW PROCESS OF

AMALGAMATION OF

GOLD AND SILVER ORES;

By BARON BORN*.

THE amalgamation of gold and ſilver ores in large operations, as well as ſmaller aſſays, requires the following diſtinct operations.

* Extracted from Mr. Raſpe's tranſlation of Baron Born's work on this ſubject.

(a) Stamp-

(a) Stamping, grinding and fifting.

(b) Calcination, and repeated grinding and fifting.

(c) Trituration.

(d) Washing of the refiduum.

(e) Eliquation of the amalgama.

(f) Heating of the fame.

(g) Diftillation of the quickfilver preffed from the amalgama.

(h) Refining of the heated quickfilver; and laftly

(i) Management, ufe, and refining of fuch refidua as ftill appear to contain fome of the nobler metals.

Stamping, grinding and fifting.

By thefe operations the picked ores, reguli, black copper, and reguline mixtures *(fpeifs)* of metals and femi-metals are reduced into fine powder, and their furfaces being thus increafed, they mix and calcine better with the common or rock-falt which is added to them; otherwife, the calcining fire and the air could not act fufficiently on the groffer particles, nor could the vitriolic and

marine acids * properly penetrate them, or a perfect desulphuration and decomposition of such substances be brought on, in which the gold and silver particles are disguised.

These operations are performed at Glasshutte, near Schemnitz, in Hungary, in *dry* stamps and mills; but at Joachimsthal, in Bohemia, *wet* stamps have been substituted, by which means the loss of dust, unavoidable in the dry grinding, and also the injury otherwise done to the health of the workmen, are prevented.

Calcination.

Sulphur can be expelled from ores in open fire and in closed vessels but imper-

* The vitriolic acid is produced in the act of calcination, by the decomposition of the sulphur contained in the ores, and the marine acid proceeds from the common salt which is purposely added to the ores, and which is decomposed by the vitriolic acid. A Glauber's salt is formed by the union of the last mentioned acid with the basis of the common salt; and the marine acid, thus set at liberty, attacks, dissolves and unites with the earthy matter, and also with the imperfect metals of the ores, by which means the particles of gold and silver are freed from their disguise or heterogeneous coverings, and laid open to, and fitted for combination with the quicksilver. This is, in few words, the *theory* of the process. *Editor.*

 fectly,

fectly, unleſs ſome proper ſubſtance be added. Thus, for example, corroſive ſublimate is uſed in order to ſeparate the ſulphur from ſome ores: in this caſe the concentrated marine acid unites with the metallic, ſemi-metallic, and ſoluble earthy particles, paſſes into the receiver with the arſenic and antimonial calx in the form of butter, and the diſengaged quickſilver ſublimates with the diſengaged ſulphur in the form of cinnabar.

From this an idea may be formed on the calcination of thoſe ores, which beſides particles of native metal, contain diſguiſed gold and ſilver, which would never be got entirely by waſhing or other mechanical contrivances, without ſome chemical aſſiſtance. This is their calcination. By fire and air, it decompoſes the ores, expels the ſulphur, puts the metallic and ſemi-metallic particles into a calciform ſtate, and freeing the noble metals from their diſguiſes, exhibits them naked in their metallic form.

If there be ſulphur enough, or even a ſuperabundance of it, calcination will produce

this

this desirable effect without any other addition. But as the vitriolic acid acts on the ores, and disengages the gold and silver particles only in proportion as it is produced from the sulphur in more or less quantity; it is safer to depend on the marine rather than the vitriolic acid: and, though common or rock-salt, added in the process of amalgamation of well-calcined ores, answers this end in some respects, yet will it serve better when mixed in proper proportions with the earthy or metallic ores before they go to the calcining fire, thus undergoing with them a similar calcination. The quantity in which it is to be added must be determined by experience. When picked and halvan ores are calcined with common salt, the sulphur and arsenic, if any, begin first to be disengaged. Part of the sulphur flies off undecomposed, a great part is converted into phlogiston and vitriolic acid, which last, uniting with the alkaline and metallic earths of the base metals and semi-metals, but in particular with the mineral alkali of the common salt,

 forms

forms with the first, different earthy and metallic (more or less soluble) neutral salts, and with the last, Glauber's salt. The marine acid, thus disengaged, begins now to act as the vitriolic acid, and is absorbed equally by the earths, and the metallic calxes.

The marine acid consequently penetrates the alkaline and metallic earths more completely than the vitriolic alone; for if there be salt enough, it decomposes all the vitriolic, earthy, and metallic neutral salts by its different elective attractions, forming therewith various new deliquescent and very soluble earthy and metallic neutral salts, by which all the disguised gold and silver particles are disengaged, laid bare, and fitted for amalgamation.

When auriferous or silvery reguline mixtures of base metals and semi-metals * (*speiss*) undergo calcination, fire and air will produce it in part, but slowly and imperfectly; whereas with common salt, or properly its

* These reguline mixtures are produced in the fusion of cobaltic, arsenical, and antimonial ores.

acid,

acid, it ſucceeds quicker and better. There is no ſulphur here, nor its acid, to decompoſe the ſalt and diſengage its acid; but common ſalt decompoſing by continued heat, its acid is as it were phlogiſticated by the action of the fire, ſeparates from its alkaline baſis, and acts immediately as a ſolvent of the metallic and ſemi-metallic particles. The elective attraction of theſe metals and ſemi-metals ſeems even to aſſiſt the decompoſition and power of the ſalt.

The different mixtures of theſe reguli (*ſpeiſs*) account for the different muriatic, metallic, and ſemi-metallic neutral ſalts. For inſtance, the reguli produced by the fuſion of the Hungarian fallow or grey copper ores, conſiſt of antimonial regulus, copper, gold and ſilver, and ſometimes alſo of ſome arſenic and iron, which in the reguli of common antimonial grey copper ores, is but in a very inconſiderable proportion. The muriatic acid, diſengaged from the ſalt, unites (gold and ſilver excepted) with the other metals and ſemi-metals,

which by calcination leave the gold and silver bare and undisguised.

The same thing happens in the calcination of auriferous or only silvery black coppers *. By the addition of common salt, the copper, iron, arsenic, and sometimes the antimonial particles are not only calcined, but also most of the antimony and arsenic is volatilized and destroyed.

The cobalt reguli produced in the treatment of arsenical cobalt and silver ores, contain very often a great quantity of bismuth. The calcination with common salt acts upon them in a similar manner; but

* Baron Born observes in a note, that Count Buffon (*Hist. nat. des Mineraux. Tome 5. page 7.*) relates, that most of the Hungarian silver ores are silvery copper ores, the richest of which he reports to contain in one hundred weight from 15 to 20 marc of silver, and a much greater proportion of copper. But this is erroneous. Hungary and Transilvania produce annually 80,000 marc of silver from silver ores, and no more than 12,000 marc of silver extracted by eliquation from silvery copper ores which commonly contain in 100 weight from four to twenty lbs. of copper, and from half to two ounces of silver. Even the black copper, let it be ever so rich, does not contain two marc per cwt. What this great naturalist says, in the same place, on the eliquation of copper, is as erroneous in every respect.

should

should they abound in bismuth, which is exceedingly fusible, this semi-metal must be taken out by eliquation before they can be well calcined, otherwise the bismuth would run, and if not wholly prevent, yet very much hinder the calcination of the other metallic and semi-metallic particles.

In calcining reguline mixtures which abound in antimony, and particularly in arsenic, it has been frequently found that more or less quantities of silver and copper are carried off by the antimonial and arsenical butter, which arises from the marine acid, chiefly when the calcining heat has been too sudden and brisk for the purpose of a quick calcination of the antimony and expeditious expulsion of the arsenic; for these volatile semi-metals acquire by the muriatic acid a much greater than their natural power to carry off and volatilize even the finest metals.

Whatever these reguline mixtures are or may be, they must have gone through the stamps and mills, and have been completely pulverised before they can be committed

to the calcining fire, which is a flaming fire kept up by the worſt of fuel; or to the calcining furnace, which conſiſts of two hearths, which, taken together, are eleven or twelve feet by five, of a grate, ſmoke and duſt chambers, communicating with a flue, and ſliding duſt-ſtoppers or duſt-dampers.

The proportion and mixture of the picked and halvan or ſtamp-ſtuff is (in Hungary) determined by and depends upon the reſpective produce of the mines and ſtamps, which is commonly two in three, and with regard to the ſilver, upon their average produce. The proportion of the ſalt is regulated and determined by the more or leſs quantity of the ſulphur of the ſaid picked and halvan ores.

Thus, for example, a whole work, parcel, or ſtem of a calcining furnace in the quick-mills in Lower Hungary, conſiſts of 30 cwt. one third or 10 cwt. of which are pulveriſed picked ore, and two thirds or 20 cwt. pulverized ſtamp or halvan-ore. Sometimes

Sometimes it conſiſts of two-fifths of the former, and three-fifths of the latter; and in that proportion it contains, upon an average, three and a half, three and three-quarters, or four ounces of ſilver per cwt. To ſuch a mixture they generally add eight per cent. of rock-ſalt *.

The calcination of ſpeiſs or reguline mixtures of baſe metals and ſemi-metals, of ſilvery black coppers, and of leadiſh ores, requires, over and above the ſalt, an addition of quick-lime, from four to ten and twelve per cent. For theſe metallic mixtures otherwiſe riſe amazingly in the calcining heat, and the ſpeiſs and black-coppers are in particular apt to turn clammy, and to leave clots in which many particles remain uncalcined; but both theſe inconveniencies are counteracted by the lime. It prevents the immediate contact of the metallic particles, and their running in the

* If pan-ſalt ſhould be uſed and prove of an inferior quality on account of fixed ammoniac (*calx ſalita*) and magneſia left in it by too briſk boiling, &c. then the proportion muſt be different. Eight per cent. of pan-ſalt will do for ſilver ore; but copper regulus frequently requires ten per cent. and black copper twelve per cent.

calcining

calcining fire: moreover, as it increases the surface of the whole mixture, the single parts of the same are more effectually acted upon, calcined, and laid bare by the fire, air, and muriatic acid.

Even the silver—regulus, and raw-stone, and the lead and copper reguli, bear a moderate addition of lime.

When the furnace is properly heated, and the doors of the dust-chambers, and the sliding-dampers, or dust-catchers of the flue are shut, the whole parcel of ore (viz. 30 cwt.) is run by wheel-barrows on the flat top of the furnace, where having been equally spread into an even surface, the proper proportion of salt and lime is sifted over it, and the whole is turned and worked with iron rakes and crooks, in every direction, until the whole is perfectly and equally mixed. Thus prepared, it is spread into a square surface, and marked into equal divisions, which, in due succession, are let down (in 8 cwt. parcels) on the upper hearth, by means of a funnel, which opens upon it through the lower vault or floor of the furnace.

nace. On this hearth it muſt be ſpread and extended equally, that the moiſture of the ſtamp or halvan-ore may be expelled, before it is ſhoved down on the lower hearth; after which, the upper hearth is immediately filled again with another quantity, that exſiccation and calcination may be performed at the ſame time, by the ſame fire.

In the calcination, the following phenomena take place: on the ſurface of the picked and halvan-ores, when brought on the lower hearth, and ſtirred, an undulating motion is obſerved, and a volatile ſulphureous acid ſmell is perceived; after which the ſulphur begins to diſengage itſelf burning, covering the whole (when the ores are very ſulphureous) with a blue flame, and flying off at laſt in the form of a whitiſh thick ſuffocating ſmoke.

Whilſt the ſulphur is thus diſengaged and decompoſed in a low or gentle fire, the vitriolic acid thence produced, decompoſes the common ſalt, combines with its mineral alkali and diſengages the marine acid, which preſently unites with the alkaline and metallic

tallic earths. At this time the ore begins to clot, to rise, extend and increase in bulk and surface. It begins to look like wet ashes, and to diffuse a mixed sulphureous saline acid smell, which proceeds from a lighter whitish or greyish blue smoke flying off from the surface.

From this instant the fire and furnace may be kept a little brighter, yet the ore must be continually stirred and turned over from one side of the furnace to the other; otherwise it would be calcined unequally, and some particles would remain undecomposed. If the furnace and fire were kept too bright, the sulphur, arsenic, and saline acid particles, too briskly expelled, would unavoidably carry along with them, and volatilize many other, nay even metallic particles *.

* Though the fire be ever so cautiously conducted, some particles of the ore will be carried off and lost. This chiefly happens in the first stage of the fire; for then, during the stirring of the ore, some particles are taken up and carried away by the current of rarefied air, or during the rising of the mixture, by means of the sulphur and marine acid. This may be in some measure prevented by keeping the back-door of the upper hearth shut; by avoiding to stir the mixture while it dusts, &c. &c.

When the fulphur begins to difengage, the ore changes its colour; it changes again when the calcination is over at its rifing and fubfidence. The fulphureous acid fmell difappears when the ore that had been rifing begins to fink, and the clotting ceafes, for then part of the muriatic acid flies off. On taking a ladle full of it for proof, or even on fmelling the whitifh rarefied fmoke near the back-door of the furnace, the fmell of pure marine acid, or of volatile fpirit of falt is perceived.

Moft ores and mixtures of ores, chiefly when containing a great proportion of pyrites, or when there happens to be fulphurated copper ore, fhew at this period a luminous phofphoric appearance, when fuddenly taken from the hearth and immediately examined in the dark. In a moderate heat and cold weather, they likewife fhew, during the ftirring, bright luminous fparks, flying and fcattering about like fire-work.

When the fulphur is fufficiently expelled, and part of the marine acid is gone; when the whole begins to fubfide, and the clotting

ting to be leſs; and when all the above-mentioned phenomena have appeared, then the calcination is deemed to be perfect.

The colour of the calcined ores and halvans is generally red, reddiſh grey, dark red, or red-brown, according to the proportion of the earthy and metallic particles, or of the ſulphur they contained. They are of a lighter and higher red when very earthy; brown when very coppery, or mixed with manganeſe; and yellow and yellowiſh red when lead prevails in their mixture.

Copper reguli, containing a much greater proportion of ſulphur, muſt undergo a longer calcining heat than other mixtures of ores and halvans. When mixed with lead or antimony and arſenic, they muſt be put to calcine not only with common ſalt, but alſo with a proportionate quantity of quick-lime, that the exceſs of muriatic acid may be taken from the butter of antimony and arſenic, and from the plumbum corneum which are produced during the calcination, and be abſorbed by the lime, which prevents the untoward clotting of the particles.

The

The ſame cautions muſt be obſerved in the calcination of the reguline mixtures (*ſpeiſs*) of baſe metals and ſemi-metals, and of the ſilvery black coppers; for they alſo contain a conſiderable proportion of antimony and arſenic. If not calcined with lime, along with the common ſalt, they pack at the very inſtant the regulus of antimony and arſenic is calcined and volatilized, in the form of a white very thick ſmoke, which is brought about very expeditiouſly by the diſengaged marine acid.

The reguline mixtures (*ſpeiſs*) of metals and ſemi-metals, and the black coppers containing little or no ſulphur, the common ſalt calcined with them is decompoſed by the action of the fire. The marine acid, thus ſet free, promotes their calcination, and forms with their calxes different perfect or imperfect neutral ſalts, diſengages the diſguiſed gold and ſilver particles, whilſt the mineral alkali remains free, and in a cauſtic ſtate: for, in the dry way, the marine acid leaves its alkali and combines with the metals and their calxes; but by ſub-

ſequent

ſequent ſolution in water, it returns to the alkali, forming again with it common ſalt, and conſequently lets go the metals, ſemi-metals, and earthy particles, which it held before in ſolution.

In the calcination of theſe metallic mixtures and the copper reguli, the before-mentioned luminous phoſphoric appearance does not take place; but the flame which paſſes over them affects various colours, and in particular the red and blue, both owing to volatilized particles of copper.

When antimonial ſtone, copper reguli, and reguline mixtures of metals and ſemi-metals are put to calcine, the antimony calcines firſt, forming a white calx in the flues and other paſſages; arſenical mixtures diffuſe a white ſmoke and garlic ſmell; thoſe which abound in lead and zinc (which laſt require a ſtronger and longer fire) produce ſaturnine zincous ſmoke and white flowers.

When the calcination of theſe metallic mixtures is perfected, and the remainder is cooled, their calxes appear browniſh grey or

or dark grey; and thoſe of the ſtone and copper reguli of a more or leſs ſaturated red colour, except thoſe which abound in lead.

The ſureſt ſymptoms, however, of their perfect calcination, are collectively the riſing and ſinking of the mixtures, their colour, and the acrid ſmell of the marine acid. Then only the gold and ſilver particles may be deemed to be fully diſengaged.

Sifting and grinding after calcination.

The grinding and ſifting of the ſtuff is as neceſſary after as before calcination; becauſe the ſtamp and halvan-ſtuff, which is mixed up with the picked ore, could not be ſifted or ground previous to its calcination on account of its moiſture. This further grinding and ſifting ſerves likewiſe fully to pulverize and equalize the clots of the reguline mixtures (*ſpeiſs*) of metals and ſemi-metals, and of the copper reguli.

If theſe coarſer particles were ſuffered to remain as they are, they might ſtill diſguiſe

many

many gold and ſilver particles, and guard them againſt amalgamation.

This repeated grinding and ſifting may be diſpenſed with when the whole ſtuff is of an equal ſize and ſufficiently fine; but its clots will at any rate require examination whether they be ſoluble in water or not, for thoſe of leady and metallic mixtures remain inſoluble. If ſoluble and not leaving ſharp coarſe particles between the fingers, they want neither grinding nor ſifting; but they muſt go through both theſe operations when inſoluble, and when they betray ſuch coarſe particles on being rubbed between the fingers.

Trituration, boiling and amalgamation of the calcined ſtuff.

By amalgamation, we underſtand that mechanical and chemical operation, in which, by means of quickſilver, heat, uninterrupted motion, and ſucceſſive contact of the particles,—gold and ſilver (previouſly diſengaged from their diſguiſes by calcination and pulveriſation)

verifation) are extracted from their earthy, metallic, or mineralized matrixes, and combined with quickfilver.

If the ores have been duly pulverized and calcined, the fuccefs of trituration, or amalgamation moftly depends on the proper proportions of the quickfilver and water which are added to the ftuff: it likewife depends on the goodnefs and conftruction of the ftirring apparatus, by which the whole mixture is kept in conftant motion and mutual contact. Even the degree of heat, and the quicknefs of the trituration or ftirring, contribute to the quicker and perfect amalgamation.

To determine the quantity of quickfilver, the weight and bulk of the calcined ftuff are to be confidered. The lighter the ftuff, the more voluminous it will prove, and confequently the gold and filver will be the more difperfed. In this cafe the quantity of quickfilver muft be proportioned to the mafs. Thus, for example, 2 cwt. of picked ore and halvans, are more bulky than 2 cwt: of calcined copper regulus or other reguli.

 The

The former, therefore, require a larger quantity of quickſilver.

Both in ſmall and great operations, experience has determined, that an exceſs of quickſilver is never hurtful, and that on the contrary a ſcanty proportion is attended with loſſes. It may be taken in the proportion of 1 to 2, that is 1 cwt. of quickſilver to 2 cwt. of ſtuff. In this proportion it does not increaſe the coſt of waſhing and preſſing, nor is any loſs of quickſilver incurred; the full produce of noble metal is thus ſecured, and the reſiduum is left poorer.

The veſſels or boilers, in which the quickſilver and ſtuff are triturated, are of copper, of an inverted conical form, and with a concave bottom. No boiling heat required: a heat of 50° or 60° is ſufficient*.

No more water is required than what will ſoak into and make the ſtuff liquid. Exceſs of water makes the quickſilver ſink too

* *Cold* amalgamation (of which an account will be given in a note ſubjoined at the end of this paper) has been ſucceſsfully introduced in Bohemia, in place of this *warm* amalgamation in copper boilers.

faſt,

faſt, and keeps the lighter and finer particles of the metals floating on the ſurface; which of courſe prevents the perfect ſucceſs of the operation. On the other hand, too little water leaves the ſtuff too thick, which makes the ſtirring very troubleſome; moreover, the evaporation of the water ſoon forms a dry and hard cruſt on the ſides of the boiler, which is attended with loſs of ſilver in the reſiduum. Experience muſt determine the proper reſpective meaſure of water.

The ſtirring apparatus is put into motion by means of a water-wheel.

The ſtirrers or ſtirring racks (which were at firſt made of copper, but have ſince been found to anſwer better when made of wood) are circular ſegments correſponding with the ſides and bottom of the boiler.

Experience only can determine how long the reſpective ſtuffs muſt be triturated with quickſilver. It has been found that ſome ſtuff yields its gold and ſilver to the quickſilver perfectly and completely within 8 or 10 hours; other mixtures require a tritura-

tion of 12 and 15 hours. Exceſs of time or longer trituration is never hurtful; too little of it will often leſſen the produce.

Waſhing of the triturated leavings or reſiduum.

The object of the trituration already deſcribed, was to unite the gold and ſilver particles of the calcined ſtuff into an amalgama with quickſilver: the object of this waſhing, is the ſeparation of this rich amalgama from the leavings or reſiduum.

This waſhing is performed in a large tub, of a conical form, with a rake within it, contrived ſo as to be thrown into a rotatory motion, by a water-wheel or by hands; with ſide cocks for drawing off the water; and with a bottom cock for tapping off the amalgama or quickſilver.

The particles of quickſilver and amalgama kept floating in the whole liquid maſs, by the continual rotation of the rake, ſink by their gravity and collect in the concave bottom of the tub, above the cock; but the remaining ſtuff or ore and ſtony matter, being much lighter, is kept floating. When the

the whole has been ſufficiently ſtirred about in this manner with the water, the bottom cock of the waſhing-tub is opened and the quickſilver and amalgama are thereby let out; after which one (or more) of the ſide cocks (which are fixed at different heights) is opened to let out the thin liquid ſtuff*.

Eliquation of the quickſilver and amalgama.

The quickſilver triturated with rich ſtuffs is ſtrained through a kind of filtrum, for the purpoſe of bringing the gold and ſilver particles into a ſmaller compaſs, and of ſeparating them from the exceſs of quickſilver; although the whole can never be ſeparated but by fire.

This is done by means of a box, on the circular opening of which lies an iron-ring, to which is fixed a bag of linen damaſk. The quickſilver (its ſurface having been previouſly cleanſed with a ſpunge from any

* This contains, beſide Glauber's ſalt, various earthy and metallic neutral ſalts. When the lixivium is very green and coppery, the copper is afterwards precipitated from it by iron.

muddy water or ſtuff that might adhere to it) is poured, in ſmall quantities, into this bag by one perſon, whilſt another preſſes it with his hands, 'till the ball of amalgama, collecting and forming apace, does no longer yield any quickſilver. When the ball becomes too big for preſſure with two hands, it is taken out, and another is formed in the ſame manner, 'till all the quickſilver is gone through the bag. The balls of amalgama are put into a wooden box.

The quickſilver which has been ſtrained through the bag (and which always contains from 20 to 30 penny-weights of gold and ſilver per cwt.) is collected in a reſervoir under the box, and ſerves again for trituration with freſh quantities of ore*.

* It is remarkable, that the greater part of the gold and ſilver remains ſuſpended in the quickſilver, when preſſed through warm as it comes from the waſhing tub; and that when left undiſturbed to cool, the amalgama congeals and ſinks to the bottom.—In freezing cold the amalgama appears cryſtallized internally, and ſeems like antimony, to conſiſt of ſhort cryſtalline fibres, croſſing each other, and even appearing on its ſurface.

Heating

Heating and ſublimation or diſtillation of the amalgama.

The amalgama-balls, obtained by preſſing or eliquating the quickſilver, conſiſt (according to the different degrees of preſſure they underwent) of 1 part ſilver and 4, 5, or 6 parts quickſilver. This is expelled from them by fire in cloſe veſſels. It is a diſtillation *per deſcenſum*, performed in large caſt-iron pots put over each other.—The fire is kept up for 5 or 6 hours.—The heat, acting through the pots on the amalgama, volatilizes the quickſilver, which, riſing in the form of vapour, and finding no paſſage in the inverted upper-pot, is forced down into the lower one, and collects there by the way of diſtillation, being condenſed and precipitated by the coolneſs that is conſtantly kept up by cold water applied to the outſide of the lower pot, or receiver.

When no copper has been revived, and the amalgama has been perfectly heated, all the quickſilver is recovered without loſs, and the balls are white like ſilver, and moſſy on

on their surfaces. If *coppery*, they have a reddish cast, and are brownish if the copper have undergone a superficial calcination. If *leady*, which is seldom the case, they shew a dark, pearl-grey colour.

Refining and cupellation of the silver.

The amalgams, according to their coppery or leady appearance, or to their purity, are either refined by cupellation, or simply melted down, and run into ingots.

When containing no gold, they may be delivered to the mint, without further fusion or cupellation, notwithstanding their copper alloy; but if auriferous and coppery, then they must be refined, or put to cupellation, that the copper may be destroyed, and the auriferous silver be brought to the standard of 15 loth 15 grains per marc, in which it is received at the mint.

Distillation of the quicksilver separated from the amalgam by heat or pressure.

The quicksilver, separated by heat from the amalgam, contains some of the noble

metals which paſſed with it through the preſſing bag. This generally amounts to 3-4 or 1 oz. per cwt. But this quickſilver being conſtantly in hand, and always ſerving in the ſubſequent triturations, its contents of gold and ſilver need only to be aſcertained once at the annual balance of the accounts. This may be done, in ſmall aſſays, by diſtilling the quickſilver with granulated lead, in glaſs retorts; but this operation ſucceeds beſt in tubulated iron-retorts, with caſt-iron receivers filled with water, and luted to the necks of the retorts. Each of theſe is ſunk half into the furnace, with their necks much inclined into the receivers. They are filled with 2 cwt. of quickſilver, to which is added 1-2 or one pound of granulated lead. The tubulated opening of the retort, and the neck of the receiver, muſt be carefully luted with refractory clay. The fire ſhould be briſk, and the whole body of the retort be covered with the burning fuel. The quickſilver riſes up in the form of vapour, and paſſes over into the receiver,

receiver, where it is condensed, collected into drops, and falls to the bottom of the water. All the auriferous silver remains behind united with the lead, which, if it should stick to the bottom of the retort, may be melted in it by a coal-fire, and poured out into an ingot, to be afterwards put to the test or cupellation.

Further treatment and use of the triturated residua which have gone through the process of amalgamation.

The residua commonly contain some gold and silver, more or less, according as they were well or badly pulverized, calcined, sifted, triturated and washed. If the residuum should contain more than 1 oz. per cwt. and raw uncalcined particles appear in the same, it will be adviseable to calcine it once more with an addition of 4 per cent. of salt, and to let it undergo a second amalgamation. If it should be of an equal size and perfectly calcined, it should be mixed up with new stuff, or triturated once more by itself.

If,

If, on the contrary, the refiduum is filvery in confequence of the imperfect wafhing and feparation of the quickfilver and amalgam, it muft be wafhed over again more abundantly diluted with water.

The lixivia containing copper are precipitated by iron.

(Note refpecting the *cold* amalgamation mentioned at p. 84.)

Confidering the complex apparatus for the *warm* amalgamation, the wear and lofs of the copper-boilers, the unequal produce, and the expence of firing (all which are now avoided) the *cold* amalgamation is, as Mr. Rafpe obferves, a noble improvement of the procefs. It was what Baron Born always aimed at, though his attempts were unfuccefsful. Mr. Gellert, at Freyberg, firft fucceeded in it, ufing wooden cylindrical churns with perpendicular piftons, laid over with copper-fheeting, which, by a quick motion up and down, produce a ftronger trituration than the rotatory horizontal motion of barrels, and at the fame time prevent the poffibility of producing fublimate or mercurius dulcis, by the excefs of marine acid acting upon the quickfilver, to which that acid has lefs affinity than to copper. His very firft experiment was attended with uncommon fuccefs; for, by cold churning, he extracted the filver from pulverized ore, which contains but $3\frac{1}{4}$ oz. per quintal, in the courfe of 16 hours, fo completely that the leavings contained but 2 dwt. (The operation may even be finifhed in 10 hours, which otherwife required 24). Upon thefe principles, the churning apparatus in wooden cylinders, has been adopted in Bohemia, with a perforated caft-iron pifton, which by a crank motion, moves quickly up and down. Though the whole is put in cold, yet at the end of the operation, it heats in confequence of the quick trituration and motion of the piftons.

At Freyberg this *cold* amalgamation is performed in a mill which turns 8 large barrels, each holding 10½ quintals of ore. The ores are dressed to contain 4 oz. per quintal, mixed with 10 per cent. of salt, and calcined and sifted in Baron Born's manner. When put into the barrels, ¼ per cent. of quicklime, and 34 lbs. of water are added, and turned briskly 2 hours, 36 turns per minute. The lime absorbs the excess of acids. To counteract the decomposition of metallic vitriols and the precipitation of silver particles (which an excess of lime might occasion) after two hours turning 2 per cent. of thin rolled iron chips, two inches square, are thrown into the barrels, and turned with the same two hours. Then the quicksilver, ½ quintal to 1 quintal of ore, is added, together with an additional 4 per cent. of iron-chips, previously coated with a little copper, by immersion in copper-water, in order to prevent the dispersion of the quicksilver, and to catch and attract its smallest particles. After these last coppery-iron-chips and the quicksilver have been added, the barrels are turned slower, at the rate of 20 or 25 turns per minute. After 12 hours turning, all the silver is extracted, except 1¼ dwt. per quintal, which cannot be further extracted by amalgamation.

XI. *CHE-*

XI. *CHEMICAL NEWS.*

THE ſtony concretions that are frequently diſcovered in the inteſtinal canal of horſes are found (*Annales de Chimie for January* 1792) to conſiſt of phoſphoric acid, vitriolic acid, volatile alkali, magneſia, argillaceous earth, calx of iron, and ſiliceous earth.

Mr. Giobert *(ibid.)* aſſerts, that, contrary to the experiments of Scheele, Bergmann, and Morveau, he has frequently obtained from calculi of the urinary bladder which he has analyſed, phoſphorated lime (earth of bones) and ſiliceous earth. He adds that he has in his collection ſome ſpecimens that are* wholly ſiliceous. He promiſes, at ſome future period, an extenſive ſet of experiments on this ſubject.

According to Mr. Bartholdi *(ibid.)* madder-root contains vitriolated magneſia.

* That appear to be?

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XII. NEW OBSERVATIONS ON

AIR AND FIRE,

AND ON THE

FORMATION OF WATER;

By Mr. SCHEELE*.

I Here take the liberty of communicating to you my new ideas reſpecting air and fire. Upon this ſubject I publiſhed, in my Treatiſe on Air and Fire, ſeveral years ago, a theory which I deduced from a courſe of experiments of my own making; and as the doctrine of fire became at that time, and has ſince continued to be, a favourite object of attention with the greateſt philoſophers in Europe, it is not to be wondered, that a

* Dr. Crell has juſtly remarked (ſee his Life of Scheele, in the 1ſt vol. of this Journal, page 18) that although the Author's reaſoning in the courſe of theſe New Obſervations diſplays great ingenuity, yet it does not carry perfect conviction with it.

Tranſlator.

number of new phenomena ſhould have been brought to light, which, however, inſtead of making againſt, rather tend to confirm my theory.

My account of the formation or ſyntheſis of heat and light from the union of empyreal air with more or leſs phlogiſton, was not much liked; yet what other concluſion could I make (when I found that a mixture of empyreal and inflammable air diſappeared after undergoing inflammation*) but that the empyreal air in union with the inflammable principle eſcaped through the glaſs, ſince on the outſide of the glaſs veſſel, in which the combuſtion took place, nothing could be perceived but heat and light? Moreover, on performing the combuſtion in cloſe veſſels over water, I could not perceive, though I repeated theſe experiments in various ways, any difference or alteration in the water. Hence I never once thought of weighing the reſiduum after the combuſtion of phoſphorus; for I concluded that it

* Treatiſe on Air and Fire, § 45, 46.

would

would probably weigh leſs, but by no means more, than the phoſphorus that was employed, ſince ſo much of the empyreal air appeared to be loſt, or to fly off in the act of combuſtion. Mr. Lavoiſier, however, found that the acid reſiduum gained in weight as much as equalled the weight of the empyreal air, in which the phoſphorus was burned. Hence it ſtruck me, that this increaſe of weight might be aſcribed partly to the heat combined with the acid reſiduum, and partly to the moiſture which the empyreal air always contains; yet it appeared to me that this would hardly counterbalance the whole weight of the loſt empyreal air.

In like manner I formerly aſcribed the increaſed weight of metallic calces to the abſorbed heat. But I was not afterwards quite ſatisfied with this opinion, for ſeveral reaſons. I now find, however, as little ſatisfaction when I attempt to account for the reduction of metallic calces and the formation of ſulphur and phoſphorus, conformably to the lateſt experiments and concluſions of our greateſt philoſophers and chemiſts, men

whom I hold in the higheſt veneration. So many doubts and difficulties ſpring up in my mind, that, before I am aware of it, I get into a labyrinth, and ſcarcely know how to find my way out again.

It is only from the accurate obſervations of a Lavoiſier that I derive any aſſiſtance in this ſtate of perplexity. How frequently have I fired a mixture of inflammable and empyreal air, and how conſtantly have I obſerved a moiſture in the glaſs veſſels, as ſoon as the inflammation was over? But I concluded that empyreal air always contains humidity, and I ſuppoſed that the inflammable air, too, might take up ſome watery particles from the vitriolic acid. Nay, the very flame of the taper employed to ſet fire to the mixture, may give out moiſture, ſince the ſmoke or flame, immediately after the exploſion has taken place, is forced back again, by the external air, into the mouth of the flaſk or phial, in which the experiment is made: Hence too, the air contained in the phial, after the inflammation has taken

taken place, ſmells like the ſmoke of a candle.

Experiment I. To ſatisfy myſelf in this matter (though I do not entertain the ſmalleſt doubts reſpecting the truth of Meſſrs. Lavoiſier's and Cavendiſh's experiments) I filled a flaſk or bottle, capable of holding 16 ounces of water, with coarſely pounded pot-aſh, and after having ſhaken out three-fourths of the ſame into a bladder containing inflammable air, I corked the bottle tight. Twenty-four hours afterwards, I emptied out the remaining portion of the pot-aſh into another bladder containing empyreal air; (I had extracted this empyreal air from nitre the day before, and into the bladder which was tied to the retort I had put a handful of pounded pot-aſh) and I then introduced into the mouth of the bottle or flaſk a red-hot wire. As ſoon as the exploſion was over, I obſerved the inſide of the bottle to be covered over with moiſture, ſo as to reſemble the ſweat (as it is called) on the cold panes of the windows in warm rooms. I was now convinced that this hu-

midity muſt be combined with empyreal air as an eſſential component part; for inflammable air, on account of its lightneſs, cannot contain any; and any watery particles which theſe two kinds of air might chance to have mechanically mixed with them, is very eaſily ſeparated by means of the pot-aſh*. Meſſ. Lavoiſier and Cavendiſh weighed the water which was produced in the combuſtion of theſe two kinds of air, and found the weight of it to be exactly equal to the weight of the two airs that had diſappeared. It is very probable, however, that both kinds of air might contain ſome accidental humidity, which uniting, after the inflammation, with the water that is eſſential to the compoſition of the empyreal air, might add a little to the weight of the product; It is, moreover, poſſible, that a body

* Pot-aſh, quicklime, pyrophorus, oil of vitriol, &c. render air, in bottles cloſely corked, ſo dry, that on hanging therein a ſlip of paper written upon with ſympathetic ink of cobalt, the writing becomes green. In like manner this dry air *calcines* (or renders efloreſcent) alum, borax, Glauber's ſalt, cryſtallized mineral alkali, martial vitriol. Oil of vitriol dries the air moſt effectually and ſooneſt.

expanded

expanded into an aërial form, may have a greater weight when brought back again to a condensed or fixed state. I am further of opinion, that the *fire* which escapes must have some, though a very trifling, weight. By the term fire I mean not only the specific heat which pre-existed in these kinds of air (for this I consider to be merely accidental) but the fire which is produced by the union of the basis of the empyreal air with phlogiston. I cannot, therefore, persuade myself that water consists of the basis of empyreal air and phlogiston; for, in the above-mentioned experiments of Mr. Lavoisier this water is actually precipitated, in consequence of the union of the basis of the empyreal air (in which this water pre-existed as a component part) with phlogiston, to which the aforesaid basis has a much stronger attraction.

I consider empyreal air, therefore, as an elastic fluid, composed of an universal, unelastic saline basis *(principium salinum)* of a small quantity of phlogiston, and of a certain proportion of water.—It is upon the

ſaline principle that the ſtrong attraction of this air to phlogiſton chiefly depends, and it is to the ſame cauſe that we are principally to aſcribe the production of fire. It is, moreover, very probable, that the nitrous acid, if we could totally deprive it of water, would very much reſemble the ſaline principle. Heat and the various kinds of light are, in my opinion, nothing but ſo many combinations of this principle with more or leſs phlogiſton.

Phlogiſton is the cauſe of the elaſticity of empyreal air; all kinds of acids, too, may be rendered volatile and elaſtic by means of this principle. The warmth of the air is, as I have already ſaid, merely accidental or extraneous; for it may be increaſed or diminiſhed by the air-pump, juſt in the ſame way as a ſpunge may be filled with water and emptied of it again by compreſſion, and yet the ſpunge itſelf remains eſſentially the ſame. In like manner the elaſticity of heat and light is entirely owing to phlogiſton.

The water which it contains is the principal cauſe of the weight of empyreal air; for

for the saline principle and phlogiston cannot weigh much, since inflammable air is so light. Now, inflammable air consists of the saline principle and a large proportion of phlogiston, or, what amounts to the same thing, of heat and phlogiston. Inflammable air cannot contain water as a component part, since it may be extracted from metals by heat alone.

This being premised, let inflammable air and empyreal air be fired together. The basis or principium salinum of the latter immediately attracts the phlogiston of the former; hence heat and light are produced. The specific heat of both kinds of air is immediately set at liberty, and this *fire**, in consequence of its extreme subtilty and elasticity penetrates through the glass vessel. The water abandoned by the empyreal air and the saline principle, not being able, by

* By the word *fire* I always mean a compound of heat and light. Of these two elastic bodies, the latter contains the greatest quantity of phlogiston (Treatise on Air and Fire, § 76). Fire, therefore, is a substance composed of two elements. Hence, according to this doctrine, elementary fire is an improper expression.

reason

reaſon of its groſſer particles and weight, to paſs through the glaſs along with the fire, neceſſarily remains behind, and collects itſelf into drops.

In this way I think we may eaſily account for the other phenomena which empyreal air exhibits with phlogiſtic bodies in general, provided the following poſition be granted: viz. Acids and metallic earths have in their pureſt ſtate (a ſtate in which we probably have never yet ſeen them) a very ſtrong attraction for a certain quantity of water, when they are deprived of their phlogiſton; and, vice verſâ, theſe acids and metallic earths part with this water again, as ſoon as they recover their phlogiſton. With regard to acids, it is a fact that they are either never free from water, or, if they are in a dry or ſolid ſtate, they ſtrongly attract humidity from the air. In the caſe of the metallic earths it is not ſo eaſy to eſtabliſh this poſition; but as they may be conſidered as ſo many ſpecies of fixed acids (I need not quote in proof of this the now well known acid nature of arſenic and molybdæna) it is very

very probable that they alſo may combine very intimately with a ſmall portion of water.

According to this doctrine, which I look upon as ſomething more than a mere hypotheſis, when a bit of phoſphorus, for inſtance, is burned, the ſaline principle of the empyreal air unites with as much phlogiſton as is neceſſary to produce fire, which fire (together with the ſpecific heat which this air had previouſly abſorbed) diſappears; whilſt the water, now deſerted by the ſaline principle, joins itſelf to the acid of the phoſphorus; and hence proceeds the augmentation of weight.

Experiment II. I provided myſelf (in the manner deſcribed in Experiment I.) with a perfectly dry empyreal air, with which I filled a matraſs or phial which had been previouſly ſtuffed full with dry ſand. I afterwards introduced into the matraſs a piece of red-hot charcoal, and let it burn till it became extinct. After the whole was become cold, I did not diſcover the leaſt trace of humidity in the veſſel; but inſtead of

empyreal

empyreal air, I had now a quantity of aërial acid or fixed air. Hence it follows, that when the aërial acid unites with phlogiſton (for it is ſo united in the ſtate in which it exiſts in charcoal) it lets go its water; but when it ſeparates therefrom, and re-aſſumes its former elaſticity, it gets back again its water (which makes one of its component parts in its elaſtic ſtate) from the empyreal air.

Experiment III. I burnt a little ſulphur in a glaſs veſſel. Two hours afterwards, when the ſmoke or fumes had ſubſided, I found ſome moiſture here and there on the inſide of the veſſel.

Experiment IV. Into a matraſs capable of holding about 40 ounces of water, I poured a little oil of vitriol, and after having ſuſpended therein a ſlip of paper written upon with ſympathetic ink of cobalt, I ſtopped the mouth of the matraſs cloſely. In the courſe of half an hour, the writing became viſibly green; but notwithſtanding this, I let the matraſs remain ſo 24 hours. I then burnt a little ſulphur, in a ſmall iron ſpoon, in

in this air. On taking out the ſpoon I cloſed the mouth of the matraſs again, but did not afterwards find the leaſt moiſture. On inverting this matraſs in a veſſel of water, ſome of the water roſe up into it; a proof that the water abſorbed the vitriolic air.

Experiment V. When I repeated this experiment, and held a ſpunge wet with cold water upon the outſide of the matraſs againſt which I directed the flame of the ſulphur that was burning within, I found, when the fumes had ſubſided, ſome, though very little, moiſture depoſited on this part of the veſſel. It ſeems, therefore, to be a matter of certainty, that water enters into the compoſition of vitriolic air; and it is probable, too, that this air, as well as the aërial acid, contains as much water as an equal quantity of empyreal air.

In the calcination of metals the ſame thing takes place. The empyreal air robs them of their phlogiſton, and their earth robs the empyreal air of its water. This accounts for the increaſed weight of metal-

lic

lic calces. Now it might be expected, that in the reduction of these calces either with or without the addition of phlogiston, the water which had so intimately combined with them, should appear again; but this is not the case.

(To be continued.)

XIII. MISCELLANEOUS

REMARKS BY MR. SCHEELE,

IN LETTERS FROM HIM TO DR. CRELL.

IN perusing the 11th part of the Latest Discoveries in Chemistry, I have met with several passages upon which I shall take the liberty of making a few remarks.

Page 6. Mr. Girtanner thinks that "iron is frequently held dissolved in mineral waters without the help of an acid." In this opinion

nion he will find few diſpoſed to coincide with him; for, if a proper inveſtigation be made, the menſtruum will be eaſily diſcovered.

Page 13. "The ſolution of the dephlogiſticated earth (or calx) of iron is not affected by tincture of galls." The fault in this caſe is ſufficiently obvious. Mr. Girtanner employed too much acid in the ſolution of the ferruginous earth; we know that ink is rendered colourleſs by acids.

Page 18. An anonymous writer quotes Mr. Wiegleb, as having prepared an aurum fulminans, by precipitating gold (from its ſolution in aqua regia) with lime-water. But this experiment of Mr. Wiegleb's was not made with ſufficient accuracy; for it is certain that no aurum fulminans can be obtained without the preſence of volatile alkali. I know from my own experience that a very ſlight pittance of volatile alkali is ſufficient for the production of a weak aurum fulminans.

We ſee how a white ſaline powder is formed round the mouths (where, after frequent

quent effusion of the contents, a little of the acid always remains adhering) of the bottles in which we keep the marine and vitriolic acids: Now, if a little pot-ash is rubbed on this part of the bottles, the smell of volatile alkali is perceived; whence it appears that this white matter is a sal ammoniac. The air of the rooms in which we sit or sleep is constantly mixed with volatile alkali; we cannot, therefore, be too careful in preventing the access of such air, not only in the present instance, but likewise in all other nice experiments. A calx of gold which was not before capable of exploding, after lying uncovered in my room only a fortnight, was converted into a weak aurum fulminans; for the calx of gold has, we know, such a strong affinity to the volatile alkali, that it decomposes every kind of sal ammoniac. Hence, in my Treatise on Air and Fire, I have considered fulminating gold as a kind of salt: For, as the experiments there related prove beyond all doubt that it contains volatile alkali, we cannot refuse to place it among the number of ammoniacal

ſalts. The volatile alkali is likewiſe the cauſe of the augmentation of weight. This writer certainly has not read what I have ſaid on this ſubject; otherwiſe he would not have invented ſuch a ſtrained and far-fetched theory of the fulmination, which is principally to be aſcribed to the air that is generated by the decompoſition or deſtruction of the volatile alkali.

Page 99. I will take upon me to ſay, that air which is blown from a pair of bellows over melted nitre, is not in the leaſt degree altered; it only becomes mixed with the empyreal air which is thrown out from the nitre.

Page 177. The acetous acid in ardent ſpirits diſtilled from grain is certainly not the true cauſe of their diſagreeable taſte. If a weak ardent ſpirit be examined during its expoſure to a very intenſe cold, it will be ſeen to be of a whitiſh colour; in ſome inſtances it will let fall a white ſediment, which, when ſeparated from the ſpirit, and held over the fire in a ſilver ſpoon, melts like fat. This oil has a very offenſive

ſmell, and if diſſolved in French brandy, it communicates to it the ſame unpleaſant taſte which malt ſpirits have. Hence I conclude that this oil, which is extracted from (rye, barley, or other) grain by fermentation, is the true cauſe of the nauſeous taſte of common ardent ſpirits.

Page 231, Is mentioned Mr. Gottling's opinion, that my mercurius dulcis obtained in the humid way, is an unſafe preparation, ſince it is not turned ſufficiently black by lime water. But I can aſſure Mr. Gottling that, if it be properly edulcorated, it does not contain the ſlighteſt veſtige of ſuperfluous ſublimate. If it did, the Medical College at Stockholm would certainly have expunged it from their third edition (it was firſt admitted into the ſecond edition) of the Swediſh Pharmacopœia. And what is the black colour which the mercurius dulcis receives from lime water, but mercury ſeparated in its metallic ſtate? The lime water abſtracts the marine acid from a portion of the corroſive ſublimate; this muriated lime diſſolves the remaining portion of the

the corrosive sublimate, in the same manner as sal ammoniac and common salt do, and then the mercury becomes free. Now, as my mercurius dulcis is finer and more divided than that which is prepared according to the old method, by levigation, even though it should be triturated for a whole year—it naturally follows, that, in order to separate the mercurius vivus from it, there is required (on account of the greater number of surfaces upon which the lime water must act) a much greater quantity of lime water than in the case of the mercurius dulcis prepared by levigation. This probably is the circumstance which has been overlooked by Mr. Gottling: He did not employ a sufficient quantity of lime water.

As long as Mr. Landriani is incapable of generating other acids from the aërial acid, practical chemists will not be very ready to subscribe to his opinion, that this acid is the source of all other acids. Let us only examine his proofs, and we shall easily be led to draw opposite conclusions.

The fixed air or aërial acid which he obtains in the diſtillation of vitriolic æther, is undoubtedly owing to the decompoſition of a portion of the ardent ſpirits; the ſame thing happens if the nitrous, arſenical, or other acids are employed in this diſtillation.

The aërial acid or fixed air which he obtains in the reduction of the calx of mercury* with charcoal, evidently proceeds from the charcoal. That he previouſly expelled the fixed air from the charcoal, proves nothing; for in the diſtillation of the mercurial calx, the phlogiſton of the charcoal combines with the nitrous acid of the quickſilver, and fixed air is then immediately diſengaged: or, what amounts to the ſame thing, the pure or empyreal air which is ſeparated during this reduction, ſeizes the phlogiſton of the charcoal, and hence the fixed air which is ſeparated from this phlogiſton, neceſſarily makes its appearance.

I know that, according to an opinion which is not yet eſtabliſhed by ſufficient proofs, the aërial acid is compoſed of pure

* Red precipitate.

air

air and phlogiſton. This is Mr. Kirwan's opinion. I have no doubt that the baſis of all acids is contained in empyreal air; but with reſpect to its converſion into fixed air, I would maintain the very reverſe of what Mr. Kirwan does; viz. that in order to become fixed air it muſt loſe a part of its phlogiſton*, and in order to appear in the form of nitrous acid, it muſt be deprived of all its phlogiſton.

The proof that this pure air is converted by the electrical ſpark into fixed air, is very equivocal. The knowledge of the nature of this ſubtle matter, as well as of many other things, is reſerved for our poſterity. It would not be difficult to ſhow that the empyreal air is dephlogiſticated in this electrical proceſs. I ſet a great value upon all Mr. Kirwan's obſervations; and if I do not admit this hypotheſis of his (viz. that fixed air conſiſts of phlogiſton and pure air) it is becauſe my own experiments ſeem to prove directly the contrary.

* Mr. Scheele ſuppoſes that phlogiſton enters into the compoſition of empyreal air. See the preceding obſervations on air, &c. page 103.

He reduces calx of mercury with iron-filings, and gets fixed air, inſtead of pure air. If I am not miſtaken this experiment was firſt made by Dr. Prieſtley. I have repeated it, and have in like manner obtained aërial acid in great abundance. The air which came laſt out of the retort was inflammable. I cannot think, however, that this aërial acid was generated during the reduction; on the contrary, I believe it to have pre-exiſted in the iron. We know that iron is ſeldom, or perhaps never, free from more or leſs plumbago; which, in this diſtillation, is decompoſed or deſtroyed by the empyreal air. This air combines with the phlogiſton of the plumbago, and hence fixed air is neceſſarily diſengaged, and collected in the receiver.

But why are iron-filings always employed in ſuch experiments, and not the purer metals? I took one ounce and a half of perfectly clean copper-filings (I ſay perfectly clean, for the leaſt impurity is ſufficient for the production of fixed air) mixed the ſame with ten ſcruples of mercurial calx prepared

with nitrous acid, and distilled this mixture in a very clean small glass retort. At first there came over red vapours, which proceeded from the nitrous acid that still adhered to the mercurial calx; the quicksilver was then reduced; the air in the retort, after the apparatus was become cold, was found to be not at all increased, but rather somewhat diminished. It extinguished flame, but I did not discover the slightest vestige of aërial acid. The extinction of the flame was owing to the phlogiston which the nitrous acid communicated to the pure air in the retort. I repeated this experiment with mercurius calcinatus per se, and with the same result, except that the air in the retort was unaltered; for in this case no nitrous acid was present.

The process with the lead amalgam, from which fixed air is said to have been obtained by distillation, appears to me to be quite as exceptionable as the conclusion respecting

the origin of the fixed air which is procured in the reduction of the mercurial calx by iron-filings. I prepared a very pure amalgam from five parts lead and four quicksilver. Of this I introduced a small quantity into a new bottle, provided with a glass stopple. I shook the bottle very strongly for a few minutes, in order to collect the dust that might be in it, and then cleared out the amalgam again. (During this violent shaking something might be rubbed off from a common cork, and hence I made use of a glass stopple; for, as I have before mentioned, the slightest impurity is capable of giving a different turn to the experiment.) After this I put the remainder of the pure amalgam, which weighed one ounce and a half, into the bottle that had been cleaned out in this manner, and shook it violently for a whole hour, during which time the inside of the bottle became lined with a black coating, and the amalgam was converted into a black powder. This powder I put into a new small glass retort, which I in like manner previously cleansed by means

of a little of this powder, from any duſt that might chance to be lodged in it, and then ſubjected it to the fire. The crackling noiſe which Dr. Prieſtley noticed during the diſtillation I alſo perceived; the quickſilver was ſublimed; but I diſcovered ſcarcely any trace of fixed air: The air in the retort appeared to be ſomewhat diminiſhed, and extinguiſhed flame.

Neither does the fixed air procured by mixing nitrous air and pure air together, prove any thing; for, if the nitrous air be previouſly agitated well with milk of lime, there is not to be perceived the ſlighteſt veſtige of fixed air; but if this be not done, ſome fixed air always ſhows itſelf as ſoon as the mixture is made, and the diminution of the pure air takes place. This fixed air muſt proceed from the nitrous acid; from the metals it cannot originate. I have procured it from nitrous air obtained from all metals that are capable of yielding nitrous air.

The aërial acid obtained in diſtilling per ſe, the calces of the baſe metals, was attracted by thoſe calces from the atmoſphere, in the

the ſame manner as it is attracted by quick-lime.

The inflammable air procured from metals cannot be conſidered as pure phlogiſton. It is a mixture of phlogiſton and the matter of heat, as I have ſhown in my Treatiſe on Fire, and muſt be reckoned among the number of the ſimpler inflammable bodies, ſuch as ſulphur, phoſphorus, &c. I look upon it to be proved, that this air is formed or generated during the treatment of the metals, in the ſame manner as the inflammable air which is obtained in the diſtillation of charcoal with cauſtic fixed alkali.

If an amalgam of lead is put into a bottle of water, well ſtopped, it deprives the water of its pure air, in the courſe of a few days; as is proved by the vitriolic teſt; I mean the green precipitate which is formed, on diſſolving about a couple of grains of martial vitriol in an ounce of this water, and afterwards adding thereto two or three drops of lixivium tartari, previouſly diluted with about two drams of the ſame water. If common water is uſed for this experiment, the martial pre-

precipitate immediately becomes yellow; that is to say, it is calcined by the pure air contained in the water.

The letter which follows this (in the first vol. of Crell's Annals for 1785) is only an abstract of Mr. Scheele's experiments and observations on æther, which have been already laid before the English reader, at full length, by Dr. Beddoes, in his translation of the Author's first Essays. We therefore deem it unnecessary to insert the aforesaid letter in this collection. EDITORS.

The acid salt which Mr. Hermbstadt has observed in the juice of cherries, was discovered by myself (in the course of my late experiments upon the acid juices of the Swedish fruits*) in a variety of juices. But as I found it to be present only in very

* See the second vol. of this Journal, page 1.

small

ſmall quantity, I purpoſely omitted to make mention of it in my paper on the Acid of Fruits and Berries.

It is not tartar, though it poſſeſſes acidity, and is pretty difficult of ſolution; but conſiſts of calcareous earth ſuperſaturated with acid of apples. When burned (during which no tartarous ſmell is perceived) it yields white aſhes, which are not fixed alkali, but calcareous earth. This ſalt may be artificially prepared, by ſaturating chalk with acid of apples, or with the juice of berberries, filtrating the ſolution, and then adding thereto a large quantity of the acid of berberries or apples, and letting it evaporate ſpontaneouſly in the open air. When the liquor has acquired the conſiſtence of a ſyrup, it will ſhoot into a number of ſmall firm cryſtals.

The thick or coagulated ſubſtance which ſeparates during the ſolution of the acid of lemons in ſpirit of wine, conſiſts of a mucilaginous matter and a little limoniated neutral ſalt. According to the method which I formerly deſcribed, a pure acid of lemons

lemons may be prepared from the juice, even though it ſhould be in a ſtate of fermentation, or be covered with mould, or (as ſometimes happens) ſhould contain an admixture of acetous or vitriolic acid; and this cryſtallized acid never afterwards ſpoils. One dram of it diſſolved in one-twelfth of a kanne (a veſſel of the capacity of three cubic inches and one-third) gives a very pure and clear juice of lemons. By triturating one dram of it with three ounces of ſugar, we obtain a very delicate lemonade powder, to be uſed, for this purpoſe, in the proportion of two ſpoonfuls to a quart of ſpring water. This powder is preferable to the common lemonade cakes or lozenges, which are apt to get moiſt.

Neutral ſalts formed with the cryſtallized acid of lemons, eaſily deliqueſce.

The neutral ſalt formed with the volatile alkali, parts with its alkali by diſtillation, and the acid is deſtroyed.

The ſolution of calcareous earth in this acid, prepared without heat, holds a good

deal

deal of superfluous earth, which separates after exposure to the open air for some days, or by boiling.

Terra ponderosa united with this acid, is somewhat more soluble than limoniated calcareous earth.

Magnesia combined with the acid of lemons gives a salt which dissolves readily in water: It is not, however, crystallizable, but puts on a gummy appearance.

A saturated solution of the aluminous earth in this acid, lets fall a precipitate in the same manner as the saturated calcareous solution.

Acid of lemons dissolves no metals except iron and zinc; both which give out (during their dissolution) inflammable air. The last mentioned solution, when brought to saturation, deposits a salt of zinc.

Among the metallic solutions, that of calx of mercury in the acetous acid is precipitated (by acid of lemons) in the form of a white powder; this is also the case with acetated lead. The last mentioned precipi-

tate

tate is re-diſſolved on the addition of nitrous acid, provided the precipitate do not contain (along with the acid of lemons) vitriolic acid.

[*The remainder of Mr. Scheele's Letters ſhall appear in the next Number.*]

XIV. EXPERIMENTS ON WOLFRAM;

By PROFESSOR GMELIN.

Experiment I.

OF finely pulverized Wolfram (freed from its matrix and heterogeneous matter as thoroughly as poſſible, and of the ſpecific gravity 5,705) I took one ounce, and after triturating it well with four ounces of perfectly dry pot-

pot-aſh, I melted it in a large iron crucible, furniſhed with a lid or cover that fitted exactly. As ſoon as the whole was melted, I poured it out upon a hot iron plate. It was of a dark and dirty green colour, and readily attracted moiſture from the air. When it was become cold, I pounded it, put it into a glazed earthen veſſel, poured upon it 48 ounces of boiling water, kept it boiling for about an hour, and afterwards ſuffered it to cool. I then poured all the lixivium upon a filter of folded blotting paper. The liquor ran through clear, and there remained upon the blotting paper a black powder, which after edulcoration and drying weighed only two drams and a half*.

Experiment II. To the clear liquor thus obtained (Experiment I.) I continued to add nitrous acid, till there ceaſed to be any efferveſcence. On the very firſt addition of the acid, white clouds were produced, not unlike thoſe which take place on the addi-

* There remained in the crucible a good deal which would not pour out with the reſt, and which could not afterwards be entirely rinſed out by hot water.

tion

tion of fixed alkali to a ſolution of alum; but theſe at firſt ſoon diſappeared, eſpecially on ſhaking the veſſel: At laſt, however, the liquor became turbid throughout, without becoming clear again when ſhaken; it ſmelt ſomewhat like liver of ſulphur, and gradually depoſited a greyiſh white powder. The whole was poured upon a filter of doubled blotting paper, and the powder that remained upon the filter was edulcorated with cold water, and dried. It weighed only 22 grains, was turned yellow by aqua fortis, and bluiſh grey by diſtilled vinegar, but was not ſoluble in either of them.

Experiment III. The filtrated liquor was again made hot, and then ſet by in the cold for ſeveral days, whereby it became turbid, and depoſited a freſh ſediment. It was then filtered again. By frequent repetitions of this treatment, I conſtantly obtained, beſides a quantity of regenerated nitre in fine cryſtals, more of the above-mentioned ſediment, which in proportion as the liquor was evaporated acquired a yellower colour (probably in conſequence of the ſuperfluous ni-

trous acid becoming more and more concentrated). It was completely ſoluble in cauſtic ſpirit of ſal ammoniac, and when dried, weighed ſeven grains and a half.

Experiment IV. Upon the black inſoluble reſiduum (Experiment I.) I poured nitrous acid; an efferveſcence enſued with a ſmell of liver of ſulphur. I continued to add more acid, and endeavoured to promote its diſſolving power by heat; but the greateſt part of the black powder remained undiſſolved at the bottom of the veſſel. Its dark colour induced me to conclude it muſt contain mangaueſe; accordingly I threw into the liquor a lump of ſugar, and heated it again: The diſſolution was thereby promoted, and the nitrous acid became of a fiery red colour. I poured off the liquor, and filtrated it: There ſtill remained behind ſome of the dark coloured powder, upon which I poured ſome freſh nitrous acid, threw in a lump of ſugar, and ſubjected the veſſel to a digeſting heat. In the courſe of a few days, the nitrous acid was become of a fiery red colour, and the ſediment

ment of a light grey. I filtered off this ſolution, and added it to that which I had poured off at firſt.

Experiment V. To the whole of the filtrated liquor (Experiment IV.) I added Pruſſian lixivium or phlogiſticated alkali (carefully prepared according to Bergmann's directions) in very ſmall quantities at a time, and I did not repeat the addition of the lixivium till the liquor was become clear again, and the precipitated matter was ſettled to the bottom, which frequently did not happen till ſeveral days had elapſed. I continued to repeat this treatment till the lixivium ceaſed to produce any alteration in the liquor. The very firſt addition of the phlogiſticated alkali rendered the nitrous ſolution turbid and greeniſh, and it conſtantly became darker and darker by further additions. I now filtered the whole through two folds of blotting paper; the liquor of the firſt percolation was ſomewhat coloured; but by repeated filtrations it was, at length, rendered perfectly limpid and colourleſs.

Experiment VI. This filtrated liquor (Experiment V.) was not rendered in the least degree turbid by lixivium tartari, nor did the portion of the liquor, upon which I made this experiment, when evaporated till only a fourth part remained, yield any precipitate.

Experiment VII. The residuum upon the filter, after edulcoration and drying, weighed four drams and a half. It was of a dark blue colour, and its surface had a coppery lustre, very much like good indigo, when it is rubbed with the nail. In order to find out how much it contained of manganese, which had so clearly discovered itself in these experiments, and which, too, according to the united testimony of all modern mineralogists, is contained in wolfram, I put the whole of this residuum into a glass vessel, filled it up with distilled water, and added (after Bergmann's method) a few drops of nitrous acid. I did not chuse to add more of the acid, lest it should dissolve some of the iron also.

Experiment VIII. At the very firſt, and before heat was applied, the great number of bubbles which roſe up in the liquor gave manifeſt proofs of diſſolution. Theſe bubbles appeared in greater abundance when the veſſel was ſubjected to a digeſting heat, in which ſtate it was left for a whole day; after which the whole was poured out upon a filter of blotting paper. The reſiduum upon the filter, after edulcoration and drying, was found to weigh exactly half an ounce.

Experiment IX. I put this inſoluble reſiduum (Experiment VIII.) into a crucible, and calcined it for ten minutes, ſo as to make it red hot throughout. It became of a ſteel grey colour, weighed only one dram and a half, and was attracted by the magnet. The reddiſh colour, however, which appeared in ſeveral parts of it, ſhowed that it ſtill contained mangancſe.

Experiment X. That which remained undiſſolved by the nitrous acid in Experiment IV. conſiſted of white granules, lying in a grey jelly, and weighing, when thoroughly

dried, 52 grains and a half. The common opinion that wolfram contains tin, its general resemblance to tin ores, the circumstance of its occurring (almost exclusively) in tin mines, added to the gelatinous appearance of the above-mentioned insoluble residuum, led me to suspect it might be calx of tin: Accordingly I treated it with a flux which has been recommended for the reduction of the calx of tin; viz. I rubbed the aforesaid residuum with two drams 15 grains of crude tartar, 17 grains and a half of nitre, and 28 grains of resin, all which had been previously reduced to fine powder. I divided this mixture into three parts, and threw each separately (but not till after the immediately preceding portion had done deflagrating) into a red hot crucible, to which I afterwards applied a cover. When the whole was thrown in, and the deflagration was over, I increased the fire a little more. When the crucible was become cold, I found therein a black spungy lump, but no regulus or metallic globule.

Experiment

Experiment XI. Having pounded this maſs or lump (Experiment X.) and melted it in another freſh crucible with a ſtronger fire, I obtained a brown ſcoria, which at firſt was hard, but ſoon became ſoft and moiſt by expoſure to the air. On boiling it with ſpring water, it coloured the water, though little of it was thereby diſſolved.

Experiment XII. To the watery ſolution of the alkaline wolfram-ſcoria (Experiment XI.) I added oil of vitriol, whereupon a ſmell of liver of ſulphur was perceived, and the liquor became turbid. Being filtered through blotting paper, it left upon the filter a bluiſh grey powder, paſſed through limpid, but after ſome time acquired a ſky-blue colour. On evaporating it a little, it let fall a very fine blue ſediment. When the whole was again poured upon the blotting paper, there was left behind upon it about one grain and a half, and the liquor paſſed through perfectly colourleſs.

Experiment XIII. The inſoluble reſiduum in Experiment XI. was partly of a tremulous conſiſtence, like jelly, and partly in a

powdery form. I dried it, and melted it once more in a ſtrong fire with burnt borax and charcoal duſt, but did not thereby obtain the leaſt trace of (reduced) metal.

Experiment XIV. I put three drams four grains of pulverized wolfram into a matraſs, poured upon it ſome marine acid or ſpirit of ſalt, placed it in a ſand-bath, and ſuffered the acid to boil gently. It ſoon acquired the fiery-red colour which it always receives from iron. I let it evaporate almoſt to dryneſs, and then poured in freſh ſpirit of ſalt. This I repeated ſeveral times, till at length I got a pretty conſiderable quantity of yellow powder. After the liquor had ſtood at reſt for a ſufficient length of time, I decanted it as clear as poſſible upon a filter of unſized paper; and when the liquor had paſſed through, I lixiviated the filter firſt with diſtilled water, and then with cauſtic ſpirit of ſal ammoniac, in order to extract all the yellow powder that might chance to be upon it. After this I removed the funnel to another matraſs. To the reſiduum in the firſt matraſs I added diſtilled water, and afterwards

terwards decanted off this water upon the firſt filter. When this liquor had run through, I further lixiviated the filter with cauſtic ſpirit of ſal ammoniac, that I might not in this caſe alſo loſe any of the yellow powder. I now, too, repeatedly lixiviated the edulcorated lixivium with cauſtic ſpirit of ſal ammoniac, till the yellow colour entirely diſappeared (which happened pretty ſoon) and nothing remained but a black powder, ſuch as the wolfram was at the firſt. I poured off the ſpirit of ſal ammoniac, which was turbid and of a browniſh colour, upon a paper filter; and in order to extract all the ſoluble matter, I lixiviated not only the wolfram reſiduum, but likewiſe the filter itſelf with diſtilled water. After the clear liquor had run through, there remained upon this filter, as well as upon the former, a browniſh matter.

Experiment XV. I digeſted the inſoluble wolfram reſiduum with freſh marine acid or ſpirit of ſalt, and repeated this treatment again and again, conſtantly edulcorating the reſiduum, after the acid was decanted from it,

it, firſt with diſtilled water, and then with cauſtic ſpirit of ſal ammoniac. I continued this treatment till I had uſed up 21 ounces of marine acid, and till there remained only 19 grains of perfectly inſoluble, clear, white granules. The acid, and the water uſed for edulcoration immediately after the acid was decanted, I mixed all together (with Experiment XIV.) In like manner, I mixed together the whole of the alkaline liquor and the water uſed for edulcoration immediately after it.

Experiment XVI. The alkaline liquor (Experiment XIV. XV.) was colourleſs, and was little affected by lixivium ſanguinis (prepared according to Mr. Struve's directions, and purified with diſtilled vinegar*) by diſtilled vinegar, or by a ſolution of verdegris in the ſame. White oil of vitriol rendered it, at firſt, turbid like opal, and

* Profeſſor Struve purifies his phlogiſticated alkali with the vitriolic, or any other *mineral* acid. (Crell's Beytrage, v. 1. p. 108.) It is Mr. Weſtrumb (ibid. p. 53) that employs the acetous acid for this purpoſe.

Tranſlator.

on

on adding more of it, threw down a whitiſh (no blue was obſerved) precipitate, as did alſo the nitrous acid. The cloudineſs, however, produced by the laſt mentioned acid, totally diſappeared again on ſhaking the liquor. A few drops of the alkaline liquor added to ſaturated ſolutions of vitriol of iron, copper and zinc, threw down whitiſh flakes; which were precipitated more copiouſly from a ſolution of alum, corroſive ſublimate, and ſugar of lead in water. From a ſolution of chalk in muriatic acid, it threw down a good deal of very fine powdery precipitate. Blue litmus paper, dipped therein, acquired, after ſome time, a reddiſh colour.

Experiment XVII. On evaporating the alkaline liquor (Experiment XIV. XVI.) in a gentle heat, there ſhot forth at the bottom, ſides, and ſurface, beautiful white cryſtals, ſome of which were needle-ſhaped, others priſmatic, and others again of a granular form.

Experiment XVIII. On holding 25 aſſay pounds of this ſalt, in a ſpoon, over the flame

flame of a candle, for about a quarter of an hour, I thought I perceived a ſmell of volatile alkali. The ſalt became yellow, and after cooling was found to have loſt 5lb. in weight.

Experiment XIX. Having put this yellow matter, after Bergmann's manner, into a Heſſian crucible which had been previouſly lined with charcoal duſt, and which was afterwards filled up with charcoal powder, and covered over with another crucible, I expoſed it for two hours in a forge furnace, the heat of which was ſo great, that all the cinders round the crucible were run together or ſlagged, and the crucible itſelf was melted faſt to its pedeſtal. I broke the crucible ſome hours afterwards, but did not find any metallic button: The matter was only melted into ſmall, ſomewhat glaſſy globules, ſome of which, on being expoſed to the blow-pipe in a ſpoon with microcoſmic ſalt, yielded a pale blue globule.

Experiment XX. Theſe globules (Experiment XIX.) I put upon a ſmall cupel, covered the ſame with charcoal powder, and placed it

it under a muffle in a fire, which I had found to anſwer for cupelling ſilver. One part of it was thereby turned yellow (Experiment XVIII.); another portion was converted into a brown ſpungy lump; and the remainder was melted into a hard maſs, which conſiſted of little globules, ſome of which had more of a glaſſy, others more of a metallic appearance; and theſe laſt were of a browniſh colour, inclining to a ſteel-grey.

(To be continued.)

XV. OF

XV. OF THE INSOLUBILITY *of some* METALS *and their* CALCES IN CAUSTIC SPIRIT OF SAL AMMONIAC*;

By Dr. HAHNEMAN.

ALTHOUGH I have here made experiments only with copper, zinc, and quickſilver, yet is it highly probable that the other metals which are ſaid to be ſoluble in cauſtic ſpirit of ſal ammoniac, will be found to be as little ſoluble therein, as theſe.

* It has been objected to me by a celebrated Teacher of Chemiſtry, that in order to have given full weight to my experiments on this ſubject, I ſhould have employed volatile alkaline air inſtead of the ammoniacal ſpirit. I allow that in that caſe the cauſtic volatile alkali would have been applied in a more concentrated ſtate; but I cannot ſuppoſe that it would have produced an effect directly contrary to that of a ſtrong ammoniacal ſpirit. Beſides, I at preſent only contend, that the generally received chemical doctrine, that zinc, copper, and quickſilver, are ſoluble in cauſtic ſpirit of ſal ammoniac, is not true.

I. Mild

I. Mild or thoroughly aerated volatile alkali diſſolves, for the moſt part by means of its fixed air, ſome metals, ſuch as zinc and copper, and likewiſe ſeveral metallic calces, among which I ſhall here only inſtance the calx of mercury.

II. Spirit of ſal ammoniac, whether ſaturated in part only, or thoroughly, with fixed air, produces this effect, and this, too, in proportion to the degree of ſaturation.

III. Cauſtic ſpirit of ſal ammoniac expoſed to the open air, attracts almoſt inſtantaneouſly a little fixed air, and after a few minutes and hours it conſtantly attracts more.

IV. According as the ammoniacal ſpirit is ſaturated with fixed air, the diſſolution of the aforeſaid metals and metallic calces takes place; otherwiſe not.

Admitting theſe four poſitions to be true*, we ſhall eaſily perceive whence it has

* That the aforeſaid metals and mercurial calx are ſoluble in the aerated volatile alkali, no one has ever denied, nor can there be any doubt about it; and that the ſame effect does *not* take place in the cauſtic ammoniacal ſpirit, will be proved by what follows.

happened,

happened, that not one chemiſt (as far as I know) has ever entertained a doubt reſpecting the ſolubility of the aforeſaid metals and metallic calces in cauſtic ſpirit of ſal ammoniac, and that all of them have looked upon it as a certain fact.

I was led by ſome circumſtances firſt of all to conſider this matter as doubtful, and I had afterwards ſtrong reaſons to believe the contrary.

At firſt I took freſh burned lime, and ſhook it with my cauſtic ſpirit of ſal ammoniac, in order to deprive it of every particle of fixed air. The reſult was exactly ſuch as I expected. But as the ammoniacal ſpirit was by this treatment impregnated with a little calcareous earth, which rendered the examination of the liquor after agitation with the metals more troubleſome, I prepared my cauſtic ſpirit of ſal ammoniac myſelf, with this variation from the uſual method, that I only let 3-4ths paſs over, did not diſtill to dryneſs, applied a very gentle heat, diſtributed the diſtilled liquor*

* It did not render lime water in the leaſt degree turbid.

into

into half ounce phials, the mouths of which were very accurately closed with ground stoppers, surrounded with wax; and matters were so contrived, that the access of the atmospherical air was precluded as completely as possible throughout the whole operation.

I judged the ammoniacal spirit to be strong enough for these experiments; since each half ounce contained (as I estimated from the quantity of sal ammoniac which I obtained by precipitating *oleum martis** with the aforesaid spirit) 19 grains of caustic, dry alkali.

(A) Into an half ounce phial filled quite full with this ammoniacal spirit, I introduced (opening and stopping the phial as quickly as possible) three grains of purified and pulverized vitriol of zinc. It spread itself out, by shaking, into a white cloud. The agitation of the bottle which was well stopped, was continued for two hours. The liquor was still turbid throughout. After

* Muriated iron.

having let it ſettle, I decanted the clear liquor carefully, but quickly. Expoſed to the heat of boiling water, it evaporated without leaving any reſiduum. The china ſaucer or bowl in which the evaporation was performed, ſmelled in this, and the other trials, like caſtor. On repeating this experiment, I carefully ſaturated the liquor, after agitation and decanting, with vitriolic acid; but no turbidneſs or precipitation enſued.

Now, if cauſtic ſpirit of ſal ammoniac was in any, even the ſmalleſt, degree capable of diſſolving zinc, ſurely the 19 grains of cauſtic, dry alkali contained in the half ounce of ſpirit, would have diſſolved a ſingle grain of calx of zinc (there certainly was not more in the three grains of cryſtallized vitriol of zinc) contained in the precipitate; ſurely the turbidneſs would have diſappeared, at leaſt in part, in the courſe of two hours!

(B) I ſhook, for the ſpace of three hours, a ſcruple of newly prepared flowers of zinc in an half ounce phial full of my ammoniacal

acal ſpirit, the mouth of the bottle being all the time well cloſed with its ſtopper. The clear liquor which was afterwards quickly decanted did not give any proof, either on evaporation, or on careful ſaturation with vitriolic acid, of having diſſolved any of the zinc.

(C) With a dram of fine zinc filings agitated and treated in the ſame manner for 24 hours, the reſult was the ſame.

(D) Into another of theſe half ounce bottles I put two grains of finely pulverized vitriol of copper, and ſtopped the bottle cloſe. It ſoon fell to the bottom in the form of a clotted ſediment. I ſhook the bottle for a couple of hours. The ſediment ſpread itſelf out into a cloud, which quickly fell to the bottom. I decanted the clear liquor, and examined it both by evaporation and ſaturation; but in vain.

I repeated this experiment, letting the clear liquor that was carefully, but expeditiouſly decanted, ſtand 24 hours in a common wine glaſs, expoſed to the air; but not the leaſt turbidneſs or blue colour made its ap-

 pearance.

pearance. There was ſcarcely a grain of calx in the precipitate from the two grains of vitriol of copper.

(E) The reſult was the ſame when I agitated a ſcruple of Brunſwick green for three hours. The clear liquor examined in all the three before mentioned ways, ſhowed no marks of ſolution.

(F) With a dram of fine copper filings, ſhaken for 24 hours, the reſult was the ſame.

(G) Two grains of cryſtallized acetated quickſilver (prepared by myſelf) were agitated in vain for two hours, in one of my half ounce phials. A clot was formed, which ſpread itſelf out into a grey cloud. On evaporating the clear liquor, which was decanted carefully, but quickly, no reſiduum was obtained. There was not much above a grain of quickſilver in the metallic ſalt here employed.

In another experiment I ſaturated the clear liquor (which was decanted after it had been ſhaken for two hours) with marine acid; no milky turbidneſs enſued.

(H) Ten grains of *mercurius ſolubilis** prepared with the greateſt nicety were ſhaken for ten hours. The clear liquor, quickly but carefully decanted, was examined in all the before-mentioned ways; but it ſhowed no ſigns of having diſſolved any of the metal.

To obviate the objection, that the diſſolution could not take place, becauſe the air or gas which is produced in ſuch caſes could not get looſe; I repeated the leading experiments (A, D, G) in the following manner: After throwing in the metallic ſalts, I quickly covered the phials, which were filled up to the very top, with a piece of (writing) paper, and ſet each of them, inverted, in a bowl filled with quickſilver. Some very ſmall air bubbles were diſengaged; but none of the metallic calces were diſſolved, as I was convinced by examining the liquors in the manner before-mentioned.

Now if perfectly cauſtic ſpirit of ſal ammoniac were capable, even in the ſmalleſt

* Quickſilver precipitated from its ſolution in nitrous acid by lime water.

 degree

degree, of dissolving the aforesaid metals, surely 19 grains of caustic dry volatile alkali, dissolved in half an ounce of water, ought to have dissolved (exhibiting a solution either wholly or in part clear) a single grain (contained in the precipitates) of calx of zinc, copper, or quicksilver. Ought not, then, the solvent power (which all, even the greatest, chemists have hitherto admitted) of the caustic spirit of sal ammoniac in these cases, to be pronounced not to exist?

P. S. Mr. Lavoisier says, that "on letting caustic spirit of sal ammoniac and red precipitate stand together, a good deal of the last was dissolved; a pellicle was formed on the liquor during the evaporation." I repeated this experiment with perfectly caustic ammoniacal spirit, in bottles quite full and well stopped; but after the space of a fortnight, I did not find that the least dissolution had taken place.

XVI. ANALYSIS OF AN

ORE OF BRASS,

From PISA, in TUSCANY;

By Mr. SAGE*.

THE ancients have described two sorts of copper, the Cyprian and the Corinthian†; the former of a red, the latter of a yellow colour. Although we have been told by some authors, that the Corinthian copper was a compound of gold, silver, and copper, yet I rather take it to have been brass produced from an ore which contained zinc and copper. Perhaps that ore was similar to this of which I am now going to give the analysis.

* Journal de Physique for February, 1791.

† Aurichalcum. Ephyrëia Æra of Virgil.

Corinthian copper, on account of its ſcarcity, fetched an exorbitant price; on account of its beauty, it was preferred to gold. If it had been nothing more than a mixture of copper, gold, and ſilver, it would have been an eaſy matter to have imitated it.

In numiſmatical collections we meet with ſpecimens of ancient medals of yellow copper or braſs; but the proceſs for making this braſs is no where deſcribed in the writings of the ancients. Hence I am induced to think, that theſe medals were ſtruck, as curioſities, in Aurichalcum or Corinthian copper.

It is to the German metallurgiſts of the 13th century that we are indebted for the art of making braſs. Albertus Magnus is the firſt who has made mention of zinc as one of the ingredients in this compound; he calls it gold marcaſſite from the property which it poſſeſſes of communicating a gold colour to red copper.

Mineralogiſts have never yet made mention of any ores, in which theſe two metallic

ſubſtances were united in ſuch proportion, as to afford, on reduction, a perfect braſs.

The ore of braſs which was ſubjected to the following experiments, comes from the neighbourhood of Piſa: It is a calamine of a dirty grey colour, with glandular ſpots of a blackiſh grey blende, of a dull luſtre and foliated texture. This calamine alſo contains ſome brown ferruginous earth and white quartz; but what peculiarly diſtinguiſhes this ore, are cryſtals of calamine of a bluiſh white colour, foliated and pearly, reſembling ſteatites. Theſe cryſtals ſometimes appear in the form of elongated plates, diverging nearly after the manner of zeolite.

Of this ore of braſs I diſtilled 300 grains in a glaſs retort, placed in a reverberating furnace. I applied the pneumatic mercurial apparatus, by which means I collected ſome mephitic acid (fixed air). At the end of the operation the ore was found to have loſt one-fourth of its weight. Being afterwards roaſted, it was not perceived to give out any ſulphureous acid.

I reduced this ore by melting it with one-third of its weight of charcoal powder, and four parts of black flux. When the mixture enters into fusion, part of the zinc burns and flies off, exhibiting a beautiful blue and green flame. As soon as this ceases, I take the crucible out of the fire, and when it is become cold, I find under the scoriæ a button of brass, which is ductile, of a most beautiful colour, and in the ratio of 12lbs. in a quintal of the ore.

This brass is as fine as that which I obtain by reducing together one part of calx of copper with two parts of calamine. In this experiment I have constantly found, that the copper retains as much zinc as is necessary for its conversion into brass, provided the metallic mixture remain covered with the scoriæ.

I proceed in the following manner: I mix 50 grains of the calx of copper, which remains after the distillation of verdegris, with 100 grains of calamine, 400 grains of black flux, and 30 grains of charcoal powder. I keep this mixture in fusion till I no longer perceive

perceive any zincous flame. When the crucible is become cold, I find under the ſcoriæ a button of braſs, which weighs one-tenth more than the regulus of copper obtained from the ſame quantity of copper calx reduced with the like quantity of flux. I have repeated this experiment ſeveral times, without any difference in the reſult; whence I conclude that copper which exhibits the colour and luſtre of gold, contains only one-ſixth part of zinc. In making braſs in the great, the copper acquires an augmentation of one-fifth in weight, and hence its colour is leſs beautiful.

The bluiſh white pearly cryſtals of coppery calamine which appear on the ſurface of this ore of braſs, diſſolve readily in nitrous acid. If a little volatile alkali be added to this ſolution, it turns blue, and the precipitated metallic calces rediſſolve in this menſtruum.

The volatile alkali is alſo capable of diſſolving the coppery calamine of Piſa. Having put 24 grains of this calcined braſs ore into a ſmall matraſs with three parts of con-

crete volatile alkali diſſolved in four parts of diſtilled water, the cupreo-zincous calx was thereby diſſolved, and I found at the bottom of the veſſel only 1-24th part of quartz.

XVII. ON THE STRENGTH OF ACIDS, AND THE PROPORTION OF INGREDIENTS IN NEUTRAL SALTS;

By R. KIRWAN, Eſq.*

IN this paper we have an account of the methods which the author has contrived for removing ſome imperfections, which himſelf and others diſcovered in his former eſſays on this ſubject.

* A ſhort extract from the fourth vol. of the Tranſactions of the Royal Iriſh Academy.

§. I

§. I.

Of the Proportion of Acid in the Mineral Acids.

It appears that the ſpecific gravity of the ſtrongeſt marine acid that can eaſily be procured and preſerved is 1,196. One hundred parts of this contain about 49 of acid, whoſe ſpecific gravity is 1,500, which is therefore taken as the ſtandard of the marine acid. The quantity of ſtandard acid 1,500 in ſpirit of ſalt of inferior denſity (temperature 60 of Fahrenheit) is then calculated, and exhibited in a table.

Vitriolic acid of the ſpecific gravity of 2,000 in the temperature of 60, is taken as the ſtandard of the ſtrength of all other vitriolic acid. Then follows a table of the quantity of ſtandard vitriolic acid 2,000 in oil or ſpirit of vitriol of inferior denſity.

The ſpecific gravity of the moſt concentrated nitrous acid which Mr. Kirwan could produce was 1,5543 in the temperature of 60. It was of a yellowiſh red colour, and ſo highly phlogiſticated and volatile, that it was

was impoſſible to make accurate experiments upon it, when mixed with ſmall proportions of water; but when mixed with an equal weight, its increaſe of denſity was about 1-12th of the whole. Then follows a table of the quantity of nitrous acid whoſe denſity is 1,5543 in ſpirit of nitre of inferior denſities. Temperature 60.

§. II.

Of the Proportion of Ingredients in different Salts.

This problem, ſays the author, involves many difficulties. For ſuppoſing the proportion of acid to be given, it ſtill remains to determine the ſtrength of that acid, elſe the word preſents no definite meaning. This can be done only by a reference to ſome known ſtandard. The chemiſts, whoſe inquiries have preceded or accompanied mine, have conſidered the acid retained by neutral ſalts in a red heat as the *ſtrongeſt poſſible*. But unleſs all theſe ſalts poſſeſs the ſame power of retaining acids in a red heat, this term

term muſt have a different ſignification when applied to each, and conſequently preſent no determinate idea. Now it is well known, that different neutral ſalts poſſeſs this power in different degrees; beſides the term *red heat* is a term of great latitude, and comprehends degrees of heat very diſtant from each other.

The determination of the proportion of water in theſe ſalts, on the ſuppoſition that they all neceſſarily contain ſome, is attended with nearly the ſame difficulties. It has generally been ſuppoſed, that the weight loſt by neutral ſalts when expoſed to a red heat, expreſſed the quantity of water of cryſtallization; but it is now known that ſome of them loſe part of their acid as well as their water in that heat, to ſay nothing of the difficulty of conſtantly employing the ſame exact degree in all caſes, or if it could be employed, of ſuppoſing that they all poſſeſs the ſame power of retaining the aqueous part. Even the ancient opinion, that cryſtallization neceſſarily implies the preſence and retention of water, is deſtitute of foundation,

tion, ſince it is now known, that cryſtals may be formed in the dry way; even many of thoſe formed in the moiſt way, as moſt ſtony cryſtals, retain no perceptible quantity of the liquid in which they were formed.

Determined by theſe conſiderations, I have abandoned in a great meaſure the ancient method, and have ſubſtituted the following: Firſt, I ſaturate a known quantity of alkali, or other baſis, with an acid whoſe ſpecific gravity is known, and whoſe proportion of ſtandard is determined by the tables. I then make another ſolution of a known quantity of neutral ſalt, of the ſame ſpecies as that formed by ſaturation, and examine the ſpecific gravity of both ſolutions in the ſame temperature, adding water to the ſtronger of the two, until their denſities become equal, and thence I infer that an equal proportion of ſalt exiſts in both; but the proportion of one of them is known, therefore the proportion in the other, the weight of the whole being found, is alſo determined. Even this method is ſubject to a ſmall inaccuracy; for a ſlight exceſs of acid

acid is always left, leſt any loſs of liquor ſhould enſue from trials of ſaturation with vegetable blues; and this renders the denſity of the ſolution of the regenerated ſalt ſomewhat greater than would enſue from the proportion of ſalt it contains, beſides that in many caſes the proportion of water of cryſtallization muſt be diſcovered by expoſure to heat.

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TABLE,

Shewing the Proportion of Acid absorbed by different Bases.

100 Parts.	Vitriolic Acid Standard.	Nitrous ditto.	Marine ditto.	Vitriolic Acid, Real.	Nitrous ditto.	Marine ditto.
Pure Vegetable Alkali	92	120	115	82,1	88,3	59,3
Mineral Alkali	143	171	145,92	127,7	136	75,19
Volatile Alkali	227	327	247	202,6	240,4	127,25
Lime -	124	258,5	104,72	110,6	190	53,94
Magnesia -	194	286	215,8	173,2	210,33	111,2

TABLE,

Shewing the Proportion of Bases absorbed by the different Acid Standards.

100 Parts.	Vegetable Alkali.	Mineral ditto.	Volatile ditto.	Lime.	Magnesia.
Vitriolic Acid	108,7	70	44	80,6	56,6
Nitrous ditto	83,33	58,4	30,58	38,4	34,9
Marine ditto	87	68,5	40,48	95,4	46,3

TABLE,

Shewing the Proportion of Basis absorbed by the different real Acids.

100 Parts.	Vegetable Alkali.	Mineral ditto.	Volatile ditto.	Lime.	Magnesia.
Vitriolic Acid	121,8	78,3	49,3	90,4	57,7
Nitrous ditto	113,2	73,5	41,2	52,6	47,6
Marine ditto	168,6	133	78,5	185	89,9

T A B L E,

Shewing the Proportion of Ingredients in different Salts.

100 Parts.	Alkali.	Water of Cryſtallization.	Acid.*
Tartar Vitriolate -	55	—	45 equivalent to 50,49 ſtandard
Nitre - -	46,15	—	53,85 equivalent to 55,7 ſtandard
Salt of Sylvius -	64,2	—	35,8 equivalent to 73 ſtandard
Glauber's Salt - -	24,16	41,3	34,54 ſtandard
Cubic Nitre - -	42,35	—	57,65 equivalent to 78,33 ſtandard
Common Salt - -	53	—	47 equivalent to 77,33 ſtandard
Vitriolic Ammoniac	27,42	10,11	62,47 ſtandard
Nitrous Ammoniac -	24	—	76 equivalent to 78 ſtandard
Common Sal Ammoniac	27,62	3,89	68,49 ſtandard

* The acid here mentioned is ſometimes the ſtrongeſt, and ſometimes intermediate between the ſtrongeſt and ſtandard; but its ſtrength may always be known by its relation to the ſtandard.

100 Parts.	Earth or Metal.	Water of Cryſtallization.	Acid.
Selenite - - -	34	23	43 ſtandard
Epſom - - -	17	50	33 ſtandard
Alum - - -	29,37	44	26,63 containing 19,78 ſtandard
Vitriol of Iron - -	22,32	39	38,68 containing 29,21 ſtandard

Quantity of Real Acid in the different Standards.*

100 Parts Vitriolic Standard contain	10,71	of Water.
100 Parts Nitrous Standard	26,46	
100 Parts Marine Standard	48,5	

* By *real* acid I do not mean, ſays the author, an acid abſolutely diveſted of all water; water being, perhaps, eſſential to the acid properties of all acids, as it certainly is of ſome of them; but barely denote by that appellation, the ſtrongeſt acid known to exiſt, whether in a combined or in a ſeparate ſtate; for inſtance, vitriolic acid as it exiſts in tartar vitriolate, &c.

XVIII. EXPERIMENTS ON

MOLYBDÆNA,

WITH A VIEW TO ITS REDUCTION:

By Mr. HIELM.

(Continued.)

§. XIII.

A. IN the following experiment, the ſtrength of the fire was increaſed by placing crucibles filled with manganeſe (as before, §. XI.) round the pedeſtal that ſupported the aſſay-crucible. The diſtance between the form or air-pipe and the center of the pedeſtal amounted to four inches and a half. The bellows were loaded with four liſpounds* (56 lbs.) and the blowing was

* A liſpound contains fourteen avoirdupois pounds.

continued

continued for an hour. Several aſſays were made at once, by ſetting one crucible within another (§. IX.)

(a) Sixteen aſs of molybdænic calx were triturated with two aſs of plumbago. The mixture being made into a ball with linſeed oil, was put into a crucible of the ſecond ſize, upon a bed of charcoal duſt (braſque) and covered over with charcoal powder. In this crucible was fixed a ſmaller one, containing

(b) A ſimilar mixture placed upon a bed of charcoal duſt. In this crucible was put another ſmaller one, containing

(c) The ſame kind of mixture as the preceding; but this mixture was neither placed upon a bed of charcoal duſt, nor covered over with charcoal powder. Laſtly,

(d) Into the fourth and uppermoſt crucible was put (upon a bed of charcoal duſt, and afterwards covered over with charcoal powder) a quantity of globules, which had been obtained by fuſing platina with earth of molybdæna, and which Mr. Hielm wanted to melt down into one button. A cru-

cible of the third ſize was inverted over the whole, and luted faſt.

After the whole apparatus had remained in the forge for the ſpace of time before-mentioned, the reſults were

(a) A coherent maſs, ſo hard as to ring. It was of a yellowiſh brown colour, and when examined with a magnifying glaſs it appeared to be interſperſed with ſmall ſhining globules or drops (Tropfen) which were probably of a droſſy nature. It weighed five aſs leſs than before, ſo that it loſt about 28 per cent. in weight. It was with difficulty broken with the fingers, at the edges, where its fracture was granular, of a ſorrel colour, and interſperſed with ſhining particles, which ſeemed to have proceeded from the plumbago.

(b) This aſſay was externally of a pale yellow or flaxen colour, and harder and more compact than the preceding. Internally it was of a dark grey colour, with a dull luſtre, not unlike iron ſcoria. In other reſpects it reſembled the preceding.

(c) The

(c) The maſs adhered to the crucible, ſo as to require to be ſeparated with a knife. Externally it was of a yellowiſh colour; on the under ſide, where it adhered to the crucible, its ſurface had a metallic ſplendour. The loſs of weight was in this caſe ſomewhat greater than in the preceding, viz. about 30 per cent. owing in part to its adheſion to the crucible. Internally it reſembled (a).

(d) All the looſe globules, procured by melting platina with the molybdænic calx, had run together into one maſs, but not ſo as to afford an uniform and perfect button.

§. XIV.

B. On repeating the fuſion of the preceding aſſays, the diſtance from the form or air-canal to the middle of the pedeſtal was altered to five inches, and the bellows were loaded with five liſpounds (70lbs.) In other reſpects the arrangement of the forge was the ſame as A. §. XIII.

The crucibles (a) and (b) were dreſſed or furniſhed in the ſame manner as before, and with

with the ſame aſſays; but the aſſay which in the former experiment had been put in crucible (c) was now only laid upon the intervening ſtand*, by the ſide of crucible (b). The ſame button, which had been before melted in it, was again put in (d), upon the ſame kind of bed, and with the ſame kind of covering as before. The bellows were worked for an hour. At the end of the operation, the aſſay in

(a) Was (externally) blackiſh, with ſhining ſcales, and ſome globules of ſcoria upon its ſurface. Being ſtruck with the hammer, it ſplit in two, and was found to be (internally) of a light grey colour, with a dull metallic luſtre, ſomewhat pipy in its texture, and interſperſed with black dots or rather holes, and alſo with ſhining ſcales.

* A flat piece of old broken crucible laid over the aſſay, ſo as to fit tight, and intervening between it and the other crucible which is introduced. It is to be underſtood that theſe intermediate ſtands or bottoms were always inſerted previouſly to the placing of one crucible within another; but as they have been before deſcribed (§. IX.) in the account of the author's general mode of adjuſting the crucibles, it is deemed unneceſſary to repeat the mention of them in every ſeparate experiment.

Tranſlator.

(b) Was

(b) Was become more compact and more truly metallic; internally of a light grey colour, which, in the recent fracture, inclined ſomewhat to a yellow. It was harder and more brittle than (a). This aſſay was of a brighter colour, on its ſurface, which was ſcattered over with ſmall, round globules, that ſhone like gold.

(c) Was melted to the ſide of the crucible, and to the intervening ſtand. In ſeveral places on its ſurface it had a dull iron grey metallic appearance; was porous and ſpungy; in other places, its ſurface was black and droſſy. Internally it had a metallic appearance, but was of a porous and ſpungy texture. On one ſide it was of a darker colour, as if it had not been equally penetrated by the heat. In the part where it had adhered to the crucible and ſtand, it had a ſhining ſurface.

(d) This metallic compound was now melted down to a button, which was, externally, ſomewhat droſſy, with globules of ſcoria upon it, in ſome places. Theſe fell

off during the cooling, and brought into view a very white and ſhining ſurface.

§. XV.

C. In another experiment with the ſame aſſays, the diſtance from the air-pipe *(form)* to the center of the pedeſtal (which was an inverted crucible of a ſmaller ſize than the former one) was four inches and a half. No manganeſe was ſet in the fire-place. The bellows were loaded with 70lbs. (five liſpounds).

(a) Half of each aſſay (a, b, c) from experiment B. §. XIV. was here laid upon a bed of charcoal duſt, and covered over with a thin layer of charcoal powder.

(b) The other half of each of the three above-mentioned aſſays was put into this ſecond crucible without any addition. Their ſhape was well obſerved, that each piece might afterwards be diſtinctly recognized.

(c) The little pieces or fragments obtained in the breaking and hammering of the three aforeſaid aſſays, were wetted with water,

ter, and rolled in unrevived calx of molybdæna, of which as much was made to ſtick to them as would. Theſe fragments were then put into this third crucible, without any further addition. A crucible was inverted and luted over the whole, which was kept in the forge for an hour.

The object of this procedure was to learn whether the refractorineſs or infuſibility of this metal was owing to a deficiency or to an exceſs of inflammable or revivifying matter; and if to the latter, which appeared to Mr. Hielm to be the likelieſt cauſe of the two, whether by depriving it of part of its phlogiſton, a well fuſed metal or regulus could be obtained. How far theſe intentions were fulfilled will be ſeen by the following reſults.

(a) Were throughout black, with ſhining grey ſcales upon their ſurfaces. Theſe ſcales might have been taken for revived metallic matter, had there been no reaſon to ſuſpect that they proceeded from the plumbago. Internally they ſhewed ſome reſemblance to a metallic nature, but for bring-

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ing them to perfection in this respect, a longer continued and more intense fire seemed to be requisite.

(b) Externally of a yellowish grey colour, somewhat sonorous, hard but brittle. Internally of a pale yellow metallic appearance, of a granular and porous texture; a proof that the fire had not been applied long enough, or had not been sufficiently intense. The small piece that had been taken from the assay B. (c) seemed to have approached nearest to fusion. On one side it had a whiter metallic appearance, and a closer texture than on the other; and in two places, on its surface, it was covered with a bronze coloured scoria, under which Mr. H. thought he perceived, on examining it with a magnifying glass, that a more perfect fusion had begun to take place.

(c) Had externally a metallic appearance, more or less of a bright yellowish colour; rough and jagged. In other respects these assays resembled (b). The coating which had been given them by rolling them in calx of molybdæna, was become incorporated with

with the reſt of their ſubſtance, and revived by the ſuper-abundant phlogiſton which they contained before.

§. XVI.

The reſults of theſe and former trials were ſufficient to remove all doubts reſpecting the revivification or metallization of the molybdænic calx. It only remained to bring it into a more perfectly fuſed ſtate. This was expected to be accompliſhed by increaſing the degree of heat, which was accordingly attempted in the following experiment, in which larger crucibles were employed than in the preceding.

D. The arrangement of the forge was the ſame as in the immediately preceding experiment, except that the bellows were worked for half an hour with 70lbs. (five liſpounds) and afterwards for three-quarters with 84lbs. (ſix liſpounds).

(a) In this crucible were put ſmall pieces or fragments of all the preceding aſſays (C. §. XV.) They were previouſly moiſten-

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ed with linſeed oil, and (a) and (b) were afterwards ſprinkled over with calx of molybdæna.

(b) Contained an aſſay, of which an account will be given in §. XIX. G.

(c) Pieces of all the former aſſays moiſtened with linſeed oil, without any farther addition.

At the end of the operation no greater approaches to fuſion appeared to have been made. Some of the pieces had begun to be ſcorified on the outſide, whilſt others appeared waſted by burning, ſo as to render it probable that the degree of heat had been too great.

It was therefore reſolved to repeat this experiment in a crucible of a more moderate ſize.

§. XVII.

E. The forge was cleaned out, which, indeed, is to be underſtood as a previous ſtep in all freſh experiments. In other reſpects things were arranged in the ſame manner as before.

(a) The

(a) The bottom of this crucible was moiſtened with a ſpunge that had been dipped in water, and as much charcoal powder was afterwards ſprinkled upon it as would ſtick to it. Some of the pieces which had been put in crucible (a) in the former experiment, were now laid in this crucible without further addition.

(b) Here were put ſome pieces that had been in (a) or (c) in the preceding experiment, and alſo one piece of F. (a) G.

(c) Some reguline pieces of the ſame aſſays were dipped in water or linſeed oil, and afterwards ſprinkled over with as much calcined borax as would adhere to them.

In other reſpects this experiment was conducted in the ſame manner as D. §. XVII.

Scarce had the blaſt been applied for half an hour with 70lbs. (five liſpounds) upon the bellows, before the (outer) crucible was obſerved to begin to melt; inſomuch that after three-quarters of an hour had elapſed, it was neceſſary to ſtop the working of the bellows, leſt the contents of the whole apparatus ſhould be loſt. On taking it out of

 the

the forge, the (outer) crucible was found to be fo foftened, that it might have been preffed flat; the luting alfo had given way.

(a) Thefe pieces adhered to the melted or foftened crucible, but fhewed no figns of having undergone fufion themfelves.

(b) Shewed no marks of fufion.

(c) Thofe pieces that were imperfectly revived, were divided or penetrated by the the borax, which, however, had not become tinged, and confequently had not diffolved any portion of them. Thofe pieces that were more perfectly metallized, had become more compact, and of a white colour, but none of them had been brought into fufion.

Without referring merely to this experiment, which was imperfect both in regard to the ftrength and duration of the fire, Mr. Hielm had many reafons for believing that the calx of molybdæna cannot be made to enter into fufion by itfelf, but rather burns or waftes away.

§. XVIII.

F. In the following experiment the bellows were loaded with 70lbs. (five lispounds) and worked for one hour. In other respects things were regulated in the same manner as before.

(a) Sixteen ass of molybdænic calx were triturated with four ass of plumbago. The mixture was moistened with linseed oil, and put into the crucible without further addition.

(b) In this crucible were put 16 ass of molybdæna, mixed in the same manner with two ass of plumbago.

(c) This crucible was brasqued or lined with charcoal dust, and a piece of black revived molybdænic metal, without plumbago, was put therein, along with a few iron filings, and was afterwards covered over with charcoal powder.

The results were,

(a) A coherent but porous mass, which had taken the shape of the bottom of the

crucible, was of a black colour, with ſome ſhining ſcales. Weighed now only 18 aſs.

(b) Melted into a blackiſh brown ſpungy ſlag or ſcoria. Weighed 12 aſs, a little of it having adhered to the inner ſides of the crucible.

(c) A ſmall metallic button, externally of a brown colour, attracted by the magnet, and ſo hard under the hammer, as not to ſplit till it had received the moſt violent blows, during which it ſtruck fire. Internally it was white, granular, and ſtreaked. Of this a more particular deſcription will be given hereafter.

§. XIX.

G. The before-mentioned compoſitions of molybdænic calx and plumbago (F. a, b) were ſubjected to further experiments, in which they were not only placed in different ſituations (A. b, D. b) but the blaſt or ſtream of air was applied ſometimes in a higher, ſometimes in a lower direction, for a longer time, viz. two hours, with the

uſual

uſual weight, four or five liſpounds (56 or 70lbs.); alſo for a ſhorter time, with a ſtill greater load upon the bellows. The reſults, however, were much the ſame as before (E. b) except that there was a conſtant diminution of weight, ſo that F. (a) weighed at laſt only ten aſs, and F. (b) nine aſs, a circumſtance that muſt be further inquired into.

§. XX.

Mr. Hielm now tried what would be the reſult (in reſpect to the buſineſs of reduction) from treating the molybdænic calx with manganeſe*.

H. Accordingly ſome finely pulverized manganeſe was moiſtened with water, and ſpread over the bottom of the crucible to the thickneſs of a few lines, and whilſt the manganeſe was yet moiſt, a little charcoal powder was preſſed into it. When this was become quite dry, a bed of charcoal duſt was laid over it, and in this were put 16

* It will afterwards be ſeen that the calx of molybdæna readily unites in the fire with regulus of manganeſe.

aſs of molybdænic calx made into a paſte with linſeed oil, and afterwards covered over with charcoal powder. A cover was luted to the crucible, which was kept in the forge for half an hour, during which time the bellows were worked with 70lbs. (five liſpounds) upon them. The bottom of the crucible gave way, whereby part of the ſcoriæ, or of the melted mangaueſe ran out. Nevertheleſs there remained in the crucible a large oblong regulus that weighed 28 aſs, and reſembled the before-mentioned (F. c.) in every reſpect, as will be more particularly noticed hereafter.

§. XXI.

I. A crucible was beſmeared with manganeſe that had been treated, in the manner deſcribed in another paper, with acids. Over this was applied a bed or lining of charcoal duſt, upon which were laid ſeveral black reguli of molybdæna, together with one yellow globule, which was for the moſt part metallic. In other reſpects the arrangement was

was the ſame as in the laſt-mentioned experiment. After three-quarters of an hour's blowing, the crucible gave way (opened) on one ſide, the charcoal duſt was conſumed, and the whole was vitrified. After ſearching for ſome minutes, I at laſt found in it a reguline globule, which exactly reſembled in its ſhape the laſt-mentioned yellow regulus of molybdæna (l) that had been put in along with the other black ones. It was now become white, ſtreaked, and granular in its texture, juſt like the compound formed by the union of regulus of manganeſe with revived or reguline molybdæna.

§. XXII.

For a want of better crucibles, of which he expected a ſupply from Mr. Geijer's manufactory, and alſo on account of the injury which his health had undergone in conſequence of his expoſure to ſuch violent degrees of heat, Mr. Hielm deſiſted from proſecuting trials of this kind any farther, for

the preſent. In the mean while, he engaged in other experiments (relative to the reduction of the molybdænic calx) that were leſs laborious, and leſs injurious to the health.

K. In order to aſcertain whether the calx of molybdæna would remain fixed in the fire, when covered with charcoal duſt, in a crucible not luted, but merely covered with a looſe lid, 16 aſs thereof were laid upon a bed of charcoal duſt, and the crucible was afterwards filled up with charcoal. The bellows were loaded with 70lbs. (five liſpounds) and worked for a quarter of an hour. The incloſed globule or button was found to have loſt not more than 25 per cent. in weight, which, as the foregoing experiments ſhow, is the uſual loſs in the reduction of this calx, even in cloſe veſſels. This button was externally of a pale yellow or flaxen colour, ſonorous, and brittle. Internally it had a light grey (and ſomewhat yellowiſh) metallic appearance. In ſhort, the molybdænic calx was become revived; for which purpoſe no other treatment than this

is

is neceſſary, as, indeed, has been ſufficiently ſhown in a former part of this treatiſe*.

§. XXIII.

L. On repeating this experiment, the crucible was filled only half way up with charcoal powder, and only 42lbs. (three liſpounds) were laid upon the bellows. On examining the calx of molybdæna after a quarter of an hour's blowing upon it, it ſtill appeared black. After being expoſed to the blaſt for another quarter of an hour, it was found to have loſt 25 per cent. in weight, had a metallic appearance externally, but was internally black and friable; whence it appears that the before-mentioned (higher) degree of heat is neceſſary to its metallization. After having been blown upon for a quarter of an hour, as in K. it weighed only ten aſs.

Theſe and the foregoing experiments afford abundant proofs, that the earth of

* See the laſt number of this Journal, p. 48.

molyb-

molybdæna is of a truly metallic nature, Perhaps it is not poſſible (as has been before obſerved) to melt it down, by itſelf, to a perfect regulus or button, ſince it will rather burn to a ſlag than enter into fuſion; but this is by no means a ſufficient reaſon for depriving it of its claim to a place amongſt the metals. Platina is known to be infuſible, yet platina is reckoned as one of the noble metals; it cannot then be thought ſtrange, that an inſtance ſhould alſo occur among the ignoble metals in which the property of fuſibility is equally wanting.

§. XXIV.

If revived molybdæna *(waſſer bleymetall)* be expoſed upon a piece of charcoal to the blow-pipe, it is ſoon ſcorified on the outſide, but its inner ſubſtance remains metallic for a pretty long time, provided the experiment be made upon a tolerably large piece. When pure it melts without intumeſcence; if otherwiſe, it is a ſign that it contains an admixture of iron, mangaueſe, &c. The

calx

calx is very difficultly diſſolved by borax, unleſs it be well dephlogiſticated, in which caſe it gives a green coloured glaſs. A piece of revived molybdæna was thrown into a crucible containing melted borax, and preſſed to the bottom. After it had been blown upon (without any weight upon the bellows) for five minutes, the glaſs of borax was found to be tranſparent and colourleſs. The piece of molybdæna itſelf was little altered; its ſurface indeed was white, and it was become more brittle.

§. XXV.

The properties which the molybdænic metal ſhews when treated in the humid way will be deſcribed hereafter. This much, however, may be mentioned now, that it is diſſolved by boiling in concentrated vitriolic acid, to which it communicates a bluiſh green colour. By ſtrong and long continued boiling the colour almoſt entirely diſappears.

Remark.

Remark. Mr. Hielm thinks that no great advantage is derived from putting manganeſe in the forge in order to increaſe the heat. Thoſe experiments in which it was not, turned out equally well with thoſe in which it was, employed.

(To be continued.)

XIX. *CHEMICAL NEWS.*

MR. WESTRING* has made a great number of experiments on dying wool and ſilk with various ſpecies of lichens. His firſt memoir, which is publiſhed in the Stockholm Tranſactions, contains his experiments on the lichenes leproſi. He has found the volatile alkali *(l'ammoniaque)* to extract their colouring matter beſt: To one part of lichen he takes ten parts of lime *(chaux)* and 1-20th part of ſal ammoniac. For dying wool, an equal weight of lichen is generally ſufficient. A little common ſalt is

* Annales de Chemie for March, 1792.

ſome-

ſometimes an uſeful addition, eſpecially for ſilk, which thereby acquires a gloſs. Mordants (or ſolutions of metallic and earthy ſalts, &c.) prove hurtful by reaſon of the gummy matter which theſe vegetables contain, and which will not bear the action of acids. The colours produced from lichens vary greatly, according to the mode of extraction, the temperature of the water employed, the quality of the water, and the ſalts therein diſſolved.

1°. The colouring particles may be extracted from ſome of them merely by maceration in cold water. Silk dyed in ſuch cold infuſions is more gloſſy, and takes as durable colours as in any other way. The lichen pſeudo-corallinus, treated in this manner, gives a moſt beautiful bright yellow colour *(couleur d'aurore)*.

2°. For the greater number, however, maceration in warm water (under 25 degrees of the Swediſh thermometer) anſwers beſt; it is alſo of great uſe as a preliminary operation, in the caſe of ſuch lichens as will not give out their colouring matter, but in higher temperatures.

3°. By

3°. By maceration in hot water (between 50 and 80 degrees of the Swedish thermometer)—by digestion, light and strong colours not obtainable by any other means, are frequently extracted from the lichens: For silk this treatment answers as well as boiling.

4°. Some of them communicate their colours immediately to boiling water, but the greater number require to be previously macerated or digested.

The lichens subjected to experiment were, 1°. L. *pertusus*. 2°. L. *sanguinarius*. 3°. L. *cinereus*. 4°. L. *rugosus*. 5°. L. *ventosus*. 6°. L. *helmatoma*. 7°. L. *subcarneus*. 8°. L. *corallinus*. 9°. L. *pseudo corallinus*. 10°. L. *tartareus*. 11°. L. *parellus*. 12°. L. *subfuscus*. 13°. L. *scruposus*. 14°. L. *impressus*. 15°. L. *candelarius*. 16°. L. *carpineus*. 17°. L. *flavescens*. 18°. L. *fusco-ater*. 19°. *Byssus candelaris*. 20°. L. *ulmi*.

The colours obtained were 1°. carmelite. 2°. grey. 3°. carmelite. 4°. yellowish carmelite and nut-brown. 5°. cinnamon colour, puce colour, purple or violet, flesh colour,

&c.

&c. 6°. yellow, olive, chesnut, nut-brown, &c. 7°. carmelite, nut-brown, yellow, &c. 8°. pistachio--nut colour, yellow, carmelite, coffee colour, brown, &c. 9°. orange colour, gold colour, brown, deep olive, &c. 10°. puce colour and violet. 11°. grey. 12°. pale yellow. 13°. deep cinnamon-brown and violet. 14°. puce colour, purplish, red, and crimson, equal to that obtained from cochineal. 15°. bad carmelite, flesh colour, &c. 16°. flesh colour. 17°. yellowish carmelite. 18°. greyish yellow. 19°. yellow. 20°. bad grey.

The variety of colours frequently obtained from one and the same species of lichen, proceeded from the diversity of treatment—with water (cold, warm, boiling) only—with spirit of wine—with volatile alkali—with water impregnated with common salt—impregnated with pot-ash—with lemon juice, vinegar, &c.

Mr. Schmeisser (Phil. Trans. for 1792, Part I.) has analysed the water of Kilburn Wells, which lie about two miles from London. From 24lbs. of this water he obtained

Fixed

Fixed air - - 84 cubic inches
Hepatic air - nearly 36
Vitriolated magnesia 910 grains, equal to 2 ounces 2 drams, apothecary's weight.
Vitriolated natron - 282 grains, or 5 drams 52 grains, apothecary's weight.
Muriated natron - 60 grains, equal to 75 grains ditto.
Selenite - - 130 grains, equal to 2 drams 42 grains ditto.
Muriated magnesia - 128 grains, equal to 2 drams 42 grains ditto.
——— calcareous earth 6 grains, equal to $7\frac{1}{2}$ grains ditto.
Aërated magnesia - $12\frac{1}{2}$ grains, equal to 15 grains ditto.
——— calcareous earth 24 grains, equal to 30 grains ditto.
Calx of iron - - $3\frac{1}{8}$ equal to 4 grains ditto.
Resinous matter - 6 grains, equal to $7\frac{1}{2}$ grains ditto.

Sum $1561\frac{5}{8}$ grains, equal to medicinal weight 4 oz. 0 dram 32 grs.

XX. NEW OBSERVATIONS ON AIR AND FIRE, AND ON THE FORMATION OF WATER;

By Mr. SCHEELE.

(*Continued*)

FOR farther ſatisfaction on this ſubject, I made the following experiments:

Experiment VI. I made ſome charcoal powder red hot in a ſmall glaſs retort; and I made half an ounce of minium red hot in a crucible, thinking that this metallic calx might have attracted moiſture as it is known to attract aërial acid, from the atmoſpherical air. I added the minium while it was yet hot, to the glowing charcoal in the retort, ſhook the whole briſkly, and applied to the long neck of the retort a ſmall cold phial. The calx of lead was inſtantly revived, and a good deal of fine charcoal duſt was forced up into

the neck of the retort by the violent expansion of the fixed air; but in my ſmall receiver, which was conſtantly kept cold, I did not perceive any moiſture, nor even in the neck of the retort. Hence I infer, that the water of the calx of lead uniting with the aërial acid or fixed air, freed from phlogiſton, gave to the fixed air the properties which it is known to poſſeſs. I found the ſmall receiver almoſt entirely full of aërial acid.

With regard to the revivification, by heat alone, of the red calx of quickſilver prepared by calcination in the fire *(mercurius præcipitatus per ſe)* it might be ſuppoſed that the water ſhould be ſeparable from it in its proper form. Experience, however, ſhews the contrary. How ſhould it, indeed, be poſſible; ſince the heat, which penetrates through the retort, is decompounded by the calces of the noble or perfect metals; for heat conſiſts, as has been before mentioned, of the ſaline principle and a conſiderable proportion of phlogiſton: now, the mercurial calx combines with part of the phlogiſton

phlogiſton which the matter of heat contains, whilſt the ſaline principle, which in ſuch proceſſes always retains a little phlogiſton, unites with the water that is diſengaged from the calx; and thus empyreal air is reproduced*.

* On a curſory view, it ſeems to be incomprehenſible how the heat, which, in the calcination of quickſilver in the open air, is generated from the phlogiſton of the quickſilver and the ſaline principle of the empyreal air,—how this heat, I ſay, can afterwards reſtore to the calx its loſt phlogiſton? It is to be obſerved, however, that the revivification in this caſe, is effected by three co-operating cauſes. In the firſt place, the attraction which the calx has to the water, is very much weakened by a much greater heat than is neceſſary for its dephlogiſtication; in the next place, there is the ſtrong attraction of the calx to phlogiſton; and in the third place, we are to take into the account the affinity of the ſaline principle, half dephlogiſticated in this reduction of the calx, to the water, whereby it is tranſmuted from heat into empyreal air. Theſe three cauſes act here ſimultaneouſly, and hence the reduction neceſſarily takes place. The reduction of other metallic calces by mere heat is brought about in the ſame manner. The water which is intimately combined with them was attracted from the menſtrua in which they were diſſolved. If they are precipitated from their ſolutions by aerated alkalis, the fixed air that is combined with them receives as much water as is neceſſary to its aeriform nature, in the ſame manner as the fixed air contained in calcareous ſpar and other ſtones; for, as has been before remarked, the aerial acid (like metallic and other mineral acids) does not part with its water, unleſs when it joins itſelf to phlogiſton, as in charcoal or black-lead.

Experiment VII. When I performed the reduction of the calx of quickſilver in a retort with charcoal powder, in the ſame manner as was practiſed with minium (the mercurial calx was previouſly heated in a crucible till it gave out fumes) the charcoal powder in the retort burnt with a bright flame; but I could not perceive the leaſt moiſture in the cold receiver. Fixed air, however, it contained in abundance. To recover the water, which is the cauſe of the increaſed weight of metallic calces, in its pure ſtate again, muſt certainly be no very eaſy matter, ſince the revivification is always brought about by means of a double elective attraction—Is it to be effected by treating calx of quickſilver with filings of metals? By no means. For,

Experiment VIII. In this caſe the metals attract the water from the mercurial calx, and the phlogiſton which they part with reduces the quickſilver. Thus the metals are actually calcined, as I have found by making trial with very pure copper filings and calx of quickſilver. Or,

Experi-

Experiment IX. With ſulphur? No. I tried this alſo with as little ſucceſs. On diſtilling a very dry calx of lead (minium) with ſulphur, I obtained a quantity of vitriolic air*, in which water is preſent as a component part. I did not perceive any moiſture in the receiver. In what manner the vitriolic air is here produced, I have ſhown in my Treatiſe on Air and Fire.

Experiment X. Reſpecting the converſion of nitrous acid into empyreal air; this is a phenomenon that may be eaſily accounted for. Nitre may be alkalized by heat alone, in cloſe veſſels (ſee Experiment XI.) and during this operation a great quantity of empyreal air is extricated. When this proceſs is performed in a glaſs retort, the nitre, as long as it continues of a deep red heat, is ſeen conſtantly boiling. In half an hour's time the boiling entirely ceaſes, though the ſame degree of heat be kept up. If the nitre be examined after it is become cold, its acid will be found to be highly phlogiſticated, and may be expelled from it

* Vitriolic acid air of Dr. Prieſtley.

 by

by vegetable acids. In other reſpects, this melted nitre is ſtill a perfectly neutral ſalt*.

* The phlogiſton which the nitrous acid acquires in this experiment muſt proceed from the decompoſition of heat, in conſequence of which empyreal air is obtained. In his notes upon my Treatiſe on Air and Fire (p. 209) Mr. Kirwan ſays, that it is the fixed alkali that phlogiſticates the nitrous acid. Notwithſtanding he quotes M. Beaumé to ſhow that fixed alkali contains phlogiſton, I ſtill entertain doubts about it. This matter is not ſo clearly proved. I know not of any chemical proceſs, by which phlogiſton is ſhown to be preſent in, and ſeparable from fixed alkali. With another view, I have diſtilled at leaſt 50 times dephlogiſticated nitrous acid over calx of quickſilver, uſing freſh nitrous acid each time; and in every diſtillation I obtained, at laſt, red plogiſticated nitrous acid as well as empyreal air. The ſame effect is produced with other abſorbent earths, when treated in the ſame manner with pure nitrous acid. Ought not the alkali, as well as the mercurial calx, ſubjected to this treatment, to undergo ſurpriſing alterations if they were deprived of ſo much phlogiſton? And yet this is not found to be the caſe! It will not, then, be thought ſtrange that I ſtill perſiſt in doubting the exiſtence of phlogiſton in fixed alkalis, till it be more clearly demonſtrated.

I repeated Dr. Prieſtley's experiment for converting nitrous acid into empyreal air, or, as he terms it, dephlogiſticated air. Of a very white dephlogiſticated nitrous acid that was highly concentrated and ſmoking, I poured a ſmall quantity into a little glaſs retort; I then inſerted the tube of a new tobacco pipe into the neck of the retort, and cloſed the juncture with a luting made of white clay and ſand. When the whole was become dry, I faſtened to the other end of the tube a bladder, containing a little milk of lime. I placed the middle part of the tube on burning charcoal, and ſet the retort over a lighted lamp, by which means I kept the acid conſtantly boiling: The bladder became diſtended with empyreal air; but perceiving it to turn yellow near the pipe

from

Here, therefore, during the fusion of the nitre, the matter of heat is decompounded, the greateſt part of its phlogiſton unites with the nitrous acid, and the water of this

from the acid which came over, I took it off, and applied a phial in its place. This became immediately full of deep red vapours. I filled ſeveral bottles with theſe fumes, which continued to be red as long as a drop of acid remained in the retort. Now as the nitrous acid which I employed was perfectly white, have I not reaſon to aſk from whence the phlogiſton, in this inſtance, proceeded? No alkali was here made uſe of. Mr. Kirwan could not miſs ſeeing this redneſs, as Dr. Prieſtley made this experiment in his laboratory. Is not the matter of heat (the elementary fire of ſome, though there is an impropriety in giving the name of element to that which is manifeſtly decompoſable) in the red hot tube decompoſed by the nitrous acid? Does not a double affinity take place in this inſtance; viz. the attraction of the nitrous acid to the phlogiſton of the matter of heat, and the attraction of the ſaline principle, deſerted by the inflammable principle, to the water of the now phlogiſticated nitrous acid; and hence the empyreal air?

I likewiſe prepared inflammable air after Dr. Prieſtley's manner (viz. by making ſpirit of wine paſs through a red hot earthen tube) I found that this air contains an admixture of fixed air which may be ſeparated from it by means of lime water. This air burns with a blue flame; when mixed (and fired) with empyreal air it gives no exploſion, but leaves behind a great deal of fixed air. To the earthen tube I applied a ſmall receiver, which I kept conſtantly cool by ſetting it in a baſon of cold water. I recovered my ſpirit of wine; but, what is remarkable, it ſmelt ſomewhat like nitrous æther (for *ſalpeterluſt* read *ſalpeternaphtha)* It ſeems to be no difficult matter to account for this phænomenon: The matter of heat unites with a little of the phlogiſton of the ſpirit of wine;

acid (for nitre and all other ſalts always retain a little water even whilſt they are kept in a ſtate of red hot fuſion) joins itſelf to the ſaline principle of the matter of heat; and hence the production of pure or empyreal air.

hence inflammable air is produced. Now, we know that in every decompoſition of ſpirit of wine, eſpecially when attended with combuſtion, fixed air is produced: Conſequently, as phlogiſton is ſeparated from it in the red hot tube, fixed air muſt alſo make its appearance. The fixed air which this inflammable air yields, in conſiderable quantity, after its combuſtion proceeds from the portion of ſpirit of wine that is intimately combined with this kind of air. Juſt ſo is it with the inflammable air that is extracted from charcoal; this air, it is true, contains ſome fixed air which is ſimply mixed with it, and which may be ſeparated from it by lime water; but, if it be fired, a much greater quantity of fixed air is obtained. Hence I infer that this air volatilized or carried up with it a little of the charcoal. With regard to the æther ſmell; the production of this is explicable by my former experiments and obſervations on different kinds of æther. After the various new kinds of æther which I have prepared, I conclude that ſpirit of wine conſiſts of a ſubtle oil, which, by means of a certain quantity of heat and phlogiſton, is rendered ſoluble in water; juſt like hepatic air, which conſiſts of ſulphur combined with a little phlogiſton and heat, and which in like manner is ſoluble in water. This air is immediately decompoſed on being deprived of its ſuperfluous phlogiſton, which is eaſily effected by nitrous acid, dephlogiſticated marine acid, or acid of arſenic. With the production of æther the ſame thing happens; the ſpirit of wine is deprived of its phlogiſton by acids or mangan eſe; the heat that was combined with this phlogiſton is let looſe (for a mixture of finely levigated mangan eſe, common marine acid, and ſpirit of wine,

Experiment XI. Now, if this phlogisticated nitre be put into a coated glafs retort, and fubjected to a ftrong fire; the attraction of the nitrous acid to phlogifton is increafed; the nitre boils afrefh; and empyreal air is again produced. When this ebullition ceafes (at which time the retort generally begins to melt) there is no longer any nitre to be found; what remains is an alkali. As the faline principle feems to exift in nitrous acid in a pretty pure ftate (for nitre, treated with inflammable fubftances, exhibits the fame phænomena as empyreal air does with fuch fubftances) it goes on attracting phlogifton from the ftrong heat, till at length, with its water, which it ftill retains, it is converted into empyreal air; in which ftate

wine, boils of itfelf without having heat applied to it) and then the fubtle oil of the vinous fpirit becomes difengaged. Let this explanation be applied to the production of æther in the preparation of Dr. Prieftley's inflammable air (for I am confident that an æther is obtained if the experiment be rightly conducted, and efpecially if the tube be very long, and as much as an ell of it at leaft, be conftantly kept red hot) it is evident from the inflammable air, that the matter of heat robs the fpirit of wine of part of its phlogifton; hence the oil of the vinous fpirit becomes difengaged. Hence, too, we fee that æther may be generated without the intervention of an acid.

it

it ceaſes to be capable of uniting with the alkali; conſequently the alkali muſt be left in a cauſtic ſtate. Dr. Prieſtley's experiments further ſhew, that if nitrous acid be made to paſs through a red hot (earthen) tube, it is entirely converted into empyreal air.

XXI. MISCELLANEOUS

REMARKS BY Mr. SCHEELE,

IN LETTERS FROM HIM TO DR. CRELL,

(CONTINUED.)

I HAVE made the following remarkable obſervation. Let an oblong phial be filled to about three-fourths of its height, with white and highly concentrated nitrous acid, and be ſet inverted in a ſmall glaſs jar, likewiſe containing concentrated nitrous acid. This jar, however, muſt be ſuch, that the oblong phial may cloſe the mouth of

of it pretty exactly, ſo as to prevent the too free evaporation of the acid. The apparatus being placed in the ſun-ſhine, the acid in the ſmall phial is ſeen to ſink lower and lower every day, and in the courſe of a fortnight or three weeks it becomes emptied of its nitrous acid; but the air which is generated is pure empyreal air, and the white nitrous acid is now turned red by the phlogiſton of the ſun's rays. I think there is little doubt, that light conſiſts of empyreal air and phlogiſton, as heat alſo does, but with a leſs proportion of phlogiſton.

I repeatedly diſtilled, in the manner deſcribed by Mr. Wenzel (in his Treatiſe on the Affinity of Bodies, p. 379) ſpirit of ſal ammoniac over arſenic, in order to fix it; but in vain: it proved to be as volatile after the operation as it was before. Indeed I doubted the fact before-hand, knowing that arſenic cannot be fixed by itſelf, but by being deprived of its phlogiſton. Yet Mr. Wenzel

Wenzel ſays that after it had undegone the above-mentioned treatment, it melted before the blow-pipe to a white maſs.

I have found that calx of lead is ſoluble in water. After triturating litharge to a very fine powder, pour upon it diſtilled water, fill a bottle therewith, cork the mouth of it tight, and ſhake it frequently. After it has ſtood two or three days, and is thoroughly ſettled, decant the clear liquor, breathe two or three times upon it, or mix with it a little water impregnated with fixed air, or add to it a drop of ſpirit of vitriol; the water will turn milky, and either ceruſſe, or vitriolated lead will ſettle at the bottom.

Concerning the ſmoking oil of vitriol, I have always thought that the only difference between it and the common ſort, which does not emit fumes, conſiſted in the greater or leſs degree of concentration, and that the ſtrongeſt is that which is ſmoking; and that in order to become fluid, it muſt attract moiſture, which immediately deprives it of its elaſtic nature. The experiments which Mr.

Mr. Dollfuſs has made on this ſubject,* clearly prove that the property of emitting fumes is by no means owing to phlogiſton; and what ſhews my opinion to be ſomething more than a mere conjecture, is the circumſtance of the ſmoking acid being ſpecifically heavier than that which does not ſmoke.

XXII. EXPERIMENTS ON WOLFRAM;

BY PROFESSOR GMELIN.

(*CONTINUED.*)

Experiment XXI.

HAVING put another portion of the before-mentioned yellow powder (Experiment XVIII.) into a ſimilar apparatus, likewiſe covered over with charcoal duſt, and having ſubjected it to a heat not quite ſo ſtrong as that in Experiment XIX. I obtained

* Crell's Annals for 1785, vol. I. p. 438.

three

three different products, viz. white vitreous globules, a ſpungy matter, like that in the preceding experiment, and a hard maſs compoſed of little globules (like that in Experiment XX.) which, when viewed through a magnifying glaſs, reſembled hæmatites *(Glaſkopfe)*.

Experiment XXII. After triturating together both the yellow powder, and the ſpungy matter (Experiment XVIII. XX.) very finely, I poured upon the ſame ſome ſpirit of ſalt; ſome bubbles were immediately extricated, and continued to riſe up through the liquor for ſome time afterwards; moſt of the powder, however, remained undiſſolved at the bottom of the veſſel. The acid, it is true, was turned yellow, and, after the ſuperfluous portion of it was ſaturated with lixivium tartari, it gave on the addition of Pruſſian lixivium (phlogiſticated alkali) and infuſion of galls, ſome, though very ſlight, marks of the preſence of iron; but on adding ſolution of gold to it, and ſaturating the predominant acid with lixivium tartari, it did not ſhew the leaſt ſign of the preſence of tin.

Experi-

Experiment XXIII. To the marine acid impregnated with the metallic matter (Experiment XIV. XV.) I added an ounce more of pure acid, and then continued to drop into the whole pure lixivium tartari, till the liquor began to become turbid. I then subjected it to heat for half an hour, in a glazed earthen veſſel, letting it boil gently at the laſt; after which, I poured it out upon a filter of blotting paper. The reſiduum upon the filter was of an ochre-yellow, gave a greeniſh glaſs on expoſure to the blowpipe with phoſphoric acid, and, after edulcoration and exſiccation, weighed only ſeven grains and a half.

Experiment XXIV. The liquor (Experiment XXIII.) paſſed through the filter clear, but ſtill tinged. I now ſaturated it completely with lixivium tartari, ſo that on the laſt addition thereof it ceaſed to become turbid; a copious precipitation took place. I poured out the whole upon blotting-paper, and let the liquor paſs through. The reſiduum upon the filter, after edulcoration and exſiccation, added to the browniſh matter,

which was left upon the filter in Experiments XIV. and XV. weighed 1 dram 15 grains; it was of a liver-brown colour, and being exposed to the blow-pipe with microcosmic salt, it gave a deep yellow coloured globule.

Experiment XXV. The residuum in Experiment XXII. proved in like manner but little soluble in burnt borax when exposed, with it, to the blow-pipe. I obtained a greenish-blue globule that was partly opake, partly transparent, with a number of grey spots interspersed through it.

Hence it is seen that most of my experiments, which I shall prosecute still farther, coincide with those which have been made, and published by Messrs. de Luyart. As far as they go, they shew that Wolfram contains a peculiar metallic substance, that is mixed with iron and manganese*, the proportions of which are not always the same; they point out a great difference

* Iron and manganese, with a little tin, were discovered by Lehmann in Wolfram. See his Physikalisch-chemische Schriften. Berlin, 8vo, 1761, p. 356.

between

between it and every other metallic body, both in the more perfect (or reguline) and in the calciform state; at least it is distinguished from the rest by its specific gravity*, by its colour, by the colour of its calx, and by the colours which it communicates to salts when melted therewith to a glass; by its refractoriness in the fire; by its difficult solubility in acids, both in its calciform and in its metallic (or reguline) state; and lastly, by the readiness with which its calx combines with caustic alkalis.

Although it resembles the noble metals in its great specific gravity, in its difficult solubility in most acids, even in aqua regia itself, and in the circumstance of being uncombinable with sulphur†; yet it differs from them in being reducible to scoria by

* Which is stated by Mess. de Luyart to be 17,6. See their Chemical Analysis of Wolfram, Sect. XI. No. 4. and Sect. XIII.

† It is true that Messrs. de Luyart (Ibid. Sect. XI. No. 3.) did obtain a lump or mass by melting the yellow powder with sulphur; but as this mass weighed only 42 grains, though 100 grains of each were employed, and as moreover it did not give out a sulphureous smell when thrown upon burning coals; it can hardly be supposed that the sulphur united with it.

lead*, in parting with its phlogiston so readily, in being so easily brought to a calx, in being irreducible without the addition of substances containing phlogiston, and in being so difficult to reduce even with such addition.

From the circumstance, too, that silver loses so much of its malleability by admixture therewith †, it is apparent that this metallic substance does not belong to the class of the noble, but rather to the class of semi-metals; if, indeed, the series of experiments made by Messrs. de Luyart prove any thing, they having been made not with the perfect (or reduced) metal, but only with its calx, which was not, as, however, it should have been, rubbed together with charcoal-powder.

* At least, after melting the yellow powder with twice as much gold, and subjecting the same to cupellation, Mess. de Luyart found nothing left upon the cupel but gold. Ibid. Sect. XII. No. 1.

† De Luyart, ibid. Sect. XII. No. 3. It is also said that tin loses of its fusibility and splendour by admixture with Wolfram. Magazin der Bergbaukunde 1785. Part I. p. 125.

As

As Wolfram is known to occur only* in or near tin-mines †, and is frequently so finely interspersed through the tin-ores, as not to be separable from them by the common methods, in consequence of which the tin, in which any of it still remains after the

* Lehman, indeed (Phys. Chem. Schrift. p. 343) makes mention of wolfram got from the principality of Halberstadt; and Wallerius (Mineralogy, German translation by J. D. Denso, Berlin, 1750, p. 347. System. Mineralogiæ Holm. Vol. I. 1772. p. 331) speaks of another sort of wolfram from Westmania in Sweden, though neither of these countries is noted for tin: But perhaps the former mistook manganese for wolfram; and the testimony of the latter is the more doubtful, as other great Swedish mineralogists, for instance Linnæus and Cronstedt, have made no mention of this Swedish wolfram.

† At least this is the case in the mines of Bohemia and Saxony; for instance, at Pressniz, Platte, Schonfeld, Schlackenwald, and Zinnwald in Bohemia; at Ehrenfriedersdorf, Geyer, Altenberg, Zinnwald, Eibenstock, Langenberg, Johanngeorgenstadt and Blankenburg in Saxony: concerning which see Ferber's Essays or Contributions towards a Mineralogical History of Bohemia. Berlin, 1775, p. 51, 98, 107, and his New Collections or Observations relative to the Mineralogical History of different Countries, &c. Mittau. Vol. I. 1778, p. 187; Charpentier Mineralogical Geography of the Elector of Saxony's Territories, Leipsic, 1778, p. 161, 167, 205, 274; Born's Index Fossilium, Pragæ 1778, Vol. I. p. 48, 49, Vol. II. p. 101. That it also occurs in the Cornish tin-mines is seen by the accounts published in the Chemical Annals for 1785, No. VI. p. 546, and seq. No. X. p. 340; No. XII. p. 519; although this is unnoticed by later writers, such as Kirwan and Cullen (in his English translation of de Luyarts memoir)

 smelting,

ſmelting, is frequently much ſpoiled *; as tin-ores, though the metal that is extracted from them is lighter than many other † metals, are heavier than moſt other ores ‡; and as it has now been proved §, that theſe properties are not owing, as was formerly ſuppoſed ||, to arſenic, which riſes in fumes from theſe ores ¶ (theſe fumes proceed from the

* Lehmann, as above, p. 346.

† Namely 7, 264 (Bergmann Sciagraphia Regni mineralis Lipſ. 1782, §. 207. p. 128) conſequently leſs than that of gold, platina, ſilver, quickſilver, lead, copper, iron, biſmuth, nickel, arſenic, cobalt, and even than the new metal itſelf of which we are here treating.

‡ The ſpecific gravity of the European tin ores is, in many inſtances, 6,750; and ſeldom leſs than 5,955 or 5000. Kirwan's Mineralogy, p. 293, and ſeq.

§ Bergman (de Docimaſia Minerarum Humida, §. X. B. Opuſcul. Vol. II. p. 439. and Sciagraphia Regni Mineralis, §. 209) and Kirwan (as above, p. 294) aſſert that tin is never mineralized by arſenic. I myſelf could not perceive the leaſt arſenical ſmell from well cleanſed and finely pulveriſed zinngraupen when thrown on burning coals.

|| Namely by Wallerius (Mineralog. p. 386, 388, and Syſtem. Mineralog. Vol. II. p. 319, 322) and even by Cronſtedt (Mineralog. German Tranſlation with Brunnick's notes and additions. Leipſic, 1770, p. 197).

¶ Theſe vapours are caught in chimnies of a particular conſtruction, where they condenſe or concrete into what is called white arſenic. Ferber, New Collections, Vol. I. p. 190, 191.

quantity

quantity of arſenical pyrites with which they are in general finely interſperſed) when they are undergoing the action of the fire; ſo I thought that the metal which has lately been diſcovered in tungſten and wolfram might perhaps be likewiſe preſent in tin-ores, and that the great weight of theſe ores, as well as ſome other properties of the tin that is extracted from them, might be owing thereto.

Accordingly, I took ſome pretty large cryſtals of *zinngraupen*, and having ſeparated them as carefully as poſſible from their matrix, I reduced half an ounce of them to fine powder, and rubbed the ſame together with two ounces of pure, dry, and finely levigated potaſh. I melted the whole in a ſtrong earthen retort, poured it out, whilſt it was in a ſtate of fuſion (it was of an aſh-grey colour) and after it was become cold and ſolid, I pounded it and boiled it for half an hour with 40 ounces of ſpring water. The liquor was poured upon a filter of blotting paper, through which it paſſed clear. I dropped aqua fortis into it, till all efferveſ-

cence ceaſed; a ſlight ſmell of liver of ſulpur was emitted, and the liquor became turbid. After ſome time a white ſediment, but in very ſmall quantity, was depoſited; there was ſo little of it, that I could not examine it accurately, and I ſhould rather think it proceeded from the calcareous earth of the ſpring-water, than from any metallic matter which the alkali had diſſolved.

Upon 5 drachms of large zinngraupen cryſtals, ſeparated as completely as poſſible from their matrix, and afterwards finely pulverized, I poured as much marine acid as covered the ſame above three fingers breadth. I then applied heat, which I continued to increaſe till the acid boiled with great violence. After a good deal of the liquor had evaporated, I removed the fire, decanted the liquor as ſoon as it was become cold, poured freſh acid upon the reſiduum, boiled it again, and repeated this treatment till at length as much as a pint of acid was conſumed.

Along with the acid I had poured off a reddiſh powder, which, now that the acid

ſtood

ſtood ſtill, gradually fell to the bottom: to collect this powder, I decanted the greater part of the liquor, and poured out the remainder with the ſediment upon a filter of blotting paper.

The acid itſelf was clear and of a gold colour, was not rendered turbid by water, but was turned blue by Pruſſian lixivium (phlogiſticated alkali) and blackiſh (but not till after the predominant acid was ſaturated with lixivium tartari) by infuſion of galls.

It is evident, therefore, that ſome particles of iron were diſſolved by the acid; that there was alſo a little tin diſſolved in it, I concluded from the following experiments.

On adding a little of it to a ſolution of gold in aqua regia, no alteration, at firſt, took place; and even when I ſaturated the ſuperfluous acid with lixivium tartari, no other effect enſued, except that the liquor became turbid, and gradually let fall to the bottom a very ſmall quantity of a whitiſh powder; but the next day this powder was of a dark purple colour.

What was ſtill held diſſolved by the acid I precipitated therefrom by lixivium tartari; there was a copious precipitation of whitiſh flocculi, which after thorough exſiccation, had a greeniſh caſt, and when thrown on burning coals turned black, without giving out any arſenical ſmell; aqua regia efferveſced ſtrongly therewith, and diſſolved the greateſt part. That there were particles of iron in what the aqua regia had diſſolved appeared on the addition of Pruſſian lixivium; and that it had alſo diſſolved a little tin, I thought probable from ſome thin whitiſh flakes that were thrown down on the addition of vitriolic acid. Nevertheleſs, the matter which was left undiſſolved by the aqua regia had more of the nature of tin.

The before-mentioned red powder, which I had poured out along with the acid, I edulcorated and dried in the ſun; it weighed 15 grains. I poured upon it ſome cauſtic ſpirit of ſal-ammoniac, and let them ſtand together for a day, in a bottle that was corked tight, and ſhaken frequently.

I de-

I decanted the liquor, and dropped into it ſome nitrous acid, whereupon it became turbid, and gradually depoſited a white ſediment; but the quantity of it was ſo ſmall, that it was ſcarcely poſſible to collect it, and examine it properly.

In like manner I poured (after having edulcorated it, and dried it in the ſun) upon the larger portion of the zinngraupen which the acid had not attacked, cauſtic ſpirit of ſal ammoniac under the ſame circumſtances as in the preceding experiment; the reſult was ſimilar, except that the ammoniacal ſpirit yielded leſs precipitate.

Being convinced by theſe experiments that the marine acid is not ſtrong enough to diſſolve the calx of tin, ſo as to diſengage the metallic matter which I ſought to extract from theſe ores, I had recourſe to another acid menſtruum, which, as we are aſſured by Wenzel*, attacks tin more forcibly, and which Bergmann† has recommended for aſſaying tin-ores.

* On the Affinity of Bodies. Dreſden, 1777 [1782] p. 179, 180.

† De Minerar. docimaſia humida. §. X. B. Opuſc. Vol. II. p. 437, 438.

Accord-

Accordingly, I put into a glaſs retort, the neck of which I had broken off, ſome pulverized zinngraupen (which was not at all attacked by marine acid even with the aſſiſtance of a boiling heat) and poured upon it pure oil of vitriol, till it covered it to ſeveral fingers breadth. I then added ſpirit of ſalt, whereupon bubbles roſe up through the liquor; I continued adding ſpirit of ſalt till no more bubbles were emitted. I then ſet the whole over the fire, and let it boil gently, continuing the application of this degree of heat for ſeveral hours. The ore did not appear to be diminiſhed in ſize. I placed it again upon the fire; and this treatment I repeated ſeveral times: at length, I ſet it by in the cold, diluted it with water, and poured off the liquor cautiouſly from the ſediment. I ſaturated the decanted liquor with lixivium tartari; but, notwithſtanding the ſtrong efferveſcence, it remained perfectly clear; the next day, however, ſome white flakes ſubſided to the bottom.

Although we may not be warranted to conclude from theſe experiments that the new

new metallic ſubſtance is not preſent in zinn-graupen; yet this much we may infer, that, if it be preſent therein, it is far more difficultly ſeparable from it (by reaſon that the calx of tin with which it is united reſiſts ſo much the action of acids, and conſequently will not let itſelf be diſengaged from that ſubſtance) than it is from the calces of iron and manganeſe with which it is combined in wolfram, or from calcareous earth with which it is combined in tungſten (*Schwerſtein*)

XXIII.

XXIII. EXPERIMENTS ON

MOLYBDÆNA,

WITH A VIEW TO ITS REDUCTION:

By Mr. HIELM.

(Continued.)

§. XXVI.

TO free the earth of molybdæna from the ſulphur with which it is combined in its native ſtate (*molybdæna membranacea* of Cronſtedt*) two methods are made uſe of, viz. torrefaction under a muffle, or diſtillation to dryneſs with pure nitrous acid. The laſt mentioned method is that which Scheele adopted in his analytical experiments upon this ſubſtance. It is an eaſy and an expeditious method; whereas the roaſting is

* Molybdænum galenare of Werner. Karſten. Muſ. Leſkean. Vol. II. p. 563.

tedious

tedious and troubleſome. Moreover, in the roaſting ſome ſmall ſcales of molybdæna, that are not completely deſulphurated, are apt to be (indeed almoſt always are) enveloped in the remaining molybdænic earth, and, being converted into yellow flowers, either fly off along with the vapours, or melt and ſink into the ſaucer or cupel. Now as Mr. Scheele's method is not liable to theſe objections, we ſhould immediately give it the preference, were it not attended with other inconveniencies, of no leſs conſequence, of which he was not aware. By digeſting molybdæna with nitrous acid, and evaporating the acid from it, the ſulphur is doubtleſs deſtroyed or decompoſed; but one of its component principles, the vitriolic acid, remains for the moſt part behind with the molybdænic earth, and combines therewith, if, according to the directions, the nitrous acid be ſtrongly and repeatedly abſtracted from the molybdæna to dryneſs. In this way we obtain a fine pale yellow (flaxen coloured) earth, which Mr. Scheele has called acid of molybdæna, becauſe it poſſeſſes

poſſeſſes ſome of the properties of acids. If this earth be melted with pure fixed alkali and charcoal powder, no ſmell of hepar ſulphuris is perceived, on adding an acid to it; and hence Mr. Scheele and others concluded that the molybdænic earth thus obtained was free from vitriolic acid. It was not then known that the earth of molybdæna poſſeſſes the ſingular property of abſorbing and uniting with hepatic air, a fact with which the chemical world was not made acquainted till Mr. Pelletier publiſhed his experiments on this ſubject in the year 1788.

From what has been ſaid it ſeems to be moſt adviſeable to make uſe of both methods together; viz. firſt of all roaſt the molybdæna in a heat quickly applied, but of a moderate degree, ſo that the ſulphur may fly off in an undecompoſed ſtate, and leave the molybdæna for the moſt part free from it. Upon this roaſted molybdæna pour nitrous acid, boil them together, and diſtil off the acid not quite to dryneſs, but decant it clear off; then add freſh nitrous acid, and proceed in this manner repeatedly, till,

though

though the acid boil ſtrongly, red vapours ceaſe to appear; this is a ſign that the acid has no further action upon the molybdæna. After the laſt quantity of acid is decanted clear off, diſtilled water made hot, is to be poured in its ſtead, upon the molybdænic acid, to be boiled briſkly therewith and decanted off; after this ſhall have been repeated ſeveral times, the molybdænic earth is to be poured out upon a filter of blotting paper, and further edulcorated. After it has been exſiccated, it is to be heated to redneſs under a muffle: thus we obtain a pure earth of molybdæna, which is as white as chalk.

To promote the diſcharge of the vitriolic acid in the roaſting, I treated the molybdæna with linſeed oil, in the manner formerly (§. VI.) mentioned. But whatever might be gained by this management in other reſpects, there was this inconvenience reſulting from it, that the alkali left by the oil, joined itſelf to the vitriolic acid, and formed with it a neutral ſalt, which mixed with the earth of molybdæna, and rendered

it

it ſtill more impure. If all the reſiduous vitriolic acid could be ſaturated in this way, it would be an eaſy matter to ſeparate the neutral ſalt thus produced, by lixiviation or edulcoration. It is beſt not to employ any linſeed oil at all in the calcination, ſince it appears that the pure molybdænic calx is (thereby) converted again into molybdæna, by which means it is made even worſe than before. By melting ſuch a calx of molybdæna with other metals, we are beſt enabled to diſcover of what degree of purity it is; for, in conſequence of the vitriolic acid which is apt to adhere to it, it is not eaſy to obtain it in this manner in a ſtate of perfect purity.

Among the various methods that have been fallen upon for deſulphurating the molybdænic calx, is to be mentioned the fuſion of the molybdæna with black flux to an hepar, from which, by means of acids, the molybdænic calx, combined with hepatic air, is ſeparated, and this air is afterwards expelled by roaſting or by digeſtion with nitrous acid. Under this treatment, however, a great

great deal of the molybdænic calx is loſt, an objection ſo much the weightier, as the molybdæna itſelf is ſo ſcarce and ſo difficultly procured. The ſhorteſt and beſt method of obtaining a pure calx of molybdæna is unqueſtionably that which has been publiſhed by Mr. Pelletier, in the Journal de Phyſique; viz. to put the molybdæna at the bottom of a large crucible, to ſet a ſmaller crucible inverted over the molybdæna, to place a lid or covercle over the whole, and ſubject the apparatus for ſome hours to a red heat. Thus the air, which is neceſſary to effect the calcination, is allowed ſufficient acceſs, whilſt, on the other hand, the veſſel is ſufficiently cloſed to prevent the diſſipation or flying off of the molybdænic calx, provided the fire be properly regulated. When the operation is finiſhed, the calx of molybdæna is found partly in the form of white ſhining flowers ſublimed into the inverted crucible, and partly in yellowiſh flowers ſitting upon the yet imperfectly decompoſed molybdæna. Both kinds are to be collected, edulcorated with water, and

 expoſed

exposed to a red heat; after which they are fit for use, being thus perfectly freed from all extraneous admixture.

§. XXVII.

Whoever subjects such a calx of molybdæna to the modes of reduction described in the preceding part of this treatise, must be convinced of its metallization. We are not to refuse it a place in the class of metals, because we have not yet been able to melt it into a round button. A metal, however, should melt with other metals; and this circumstance will afford fresh grounds for considering reduced molybdæna (*wasserbleymetall, molybdænum*) as a peculiar metal, since it is a fixed maxim, that metals cannot be united with any other bodies but metals so as to retain, after the union, their leading properties.

Agreeably to this maxim, the admixture by which iron is rendered cold short, was at first supposed to be a new metal, and was named siderite (*siderum*); but it was soon afterwards discovered that this bad quality of the

the iron was not owing to a peculiar metallic body, but to a ſubſtance of a very different nature, viz. to a compound of calx of iron and phoſphoric acid. That the combination of the molybdænic metal (*molybdænum*) with other metals does not depend upon any ſuch mineralizing property, will appear from the following experiments. On this occaſion it is proper to call to mind, that ductile metals melted with brittle metals, give a brittle compound; that metals which are not ductile are by ſuch addition rendered ſtill more brittle; that ductile metals are capable of uniting with a ſmall proportion of brittle metals without loſing any of their former properties; that refractory metals may be rendered leſs difficultly fuſible by admixture with others, &c. In all caſes, however, the metallic luſtre, with the circumſtances therewith connected, are the beſt and ſureſt proofs of metallic combination.

Now, as the admixture of metals by fuſion may be effected in various ways, viz. either by taking them in the ſtate of calces,

or in the reguline ſtate, or by taking one of them in a calciform, and the other in the metallic ſtate; Mr. H. undertook to examine the affinities of molybdænum with ſome of the other metals, under theſe different circumſtances. He has choſen to begin with copper.

§. XXVIII.

I. Fuſion of calx of molybdæna with copper.

A. The arrangement in the forge was ſuch, that the centre of the pedeſtal was diſtant five inches from the mouth of the air-channel or *form* *. The bellows were worked for half an hour; no manganeſe was put among the fuel, the weight upon the bellows from the beginning to the end of the operation amounted to five liſpounds (70lbs.) Several aſſays were made at once, as in the former experiments. As before, the crucibles were marked alphabetically, and braſqued; and the introduced mixtures

* *Tuyere* of the French.

were

were covered over with a thin layer of charcoal powder.

The calx of molybdæna ufed in thefe experiments was obtained by calcination in a crucible promoted by the affufion of linfeed oil; (§. VI.) after which it was carefully roafted under a muffle, fo as to be converted into flowers, which were nicely picked out without being previoufly wafhed.

(a) In this outermoft crucible was put a mixture of 48lbs. (affay weight) of copper *(Streukupfer)* rubbed together with 16lbs. of molybdænic calx, and afterwards formed into little balls by means of linfeed oil.

(b) This crucible contained 12lbs. of the fame kind of copper, and 16lbs. of molybdænic calx.

(c) Copper 16lbs. and calx of molybdæna 16lbs. and laftly,

(d) Copper 8lbs. calx of molybdæna 16lbs.

The whole apparatus after being covered over with an inverted crucible, was made and kept red hot for fome time before the bellows were played, to allow the molybdænic

dænic calx to abſorb phlogiſton, and thereby to fit it for gradually uniting with the copper, which otherwiſe would quickly have melted down to the bottom of the crucible, without combining with any of the aforeſaid calx.

On examining the crucibles, there was found in

(a) A thin plate, with raiſed or upright edges; it was of a copper colour, interſperſed with white ſpots, and weighed 52lbs.

(b) Reſembled the preceeding, but ſeemed to have been diſpoſed to mix better in the fuſion; it weighed 44lbs.

(c) A hemiſpherical, ſmooth, and even button, which weighed 26lbs.

(d) A dark blue porous globular maſs, which weighed 26lbs.

The great loſs of weight experienced in theſe and the ſubſequent aſſays, is owing to ſmall globules of the copper getting into the braſque. On repeating the fuſion

B. The diſtance between the *form* or *tuye e*, and the pedeſtal, was leſſened. In other reſpects the arrangement was as before.

(a) In

(a) In this crucible a partition was made of a piece of old broken crucible faſtened at the bottom with luting, and in the two cavities were laid the aſſays A. (c) and (d) without further addition.

(b) Contained the ſame aſſay as in experiment A, but without braſque and charcoal-duſt.

(c) Contained the aſſay A. (a).

(d) Into this crucible, which was thinly braſqued, was put 1 quintal (=69 $\frac{1}{8}$aſs =100lbs.) of pure copper *(Streukupfer)* made into a ball with linſeed oil, and covered over with a little charcoal-duſt.

Over the whole apparatus was inverted a crucible, to ſerve as a cover. After half an hour's blowing, all the aſſays were found to have exactly the ſame weight as before; but were more ſcorified on their ſurfaces, which appeared of a bluiſh grey colour. None of them were better fuſed than before, except A. (c) which might be ſaid to be ſomewhat more run together, though it was ſtill far from being brought to a button or regulus.

In the uppermoſt crucible the copper itſelf had not been perfectly fuſed.

C. In another trial, theſe aſſays were not brought to a much more perfect fuſion. Even pure copper, which was laid in the outermoſt crucible, afforded no perfect button. But when this copper was fuſed per ſe, it melted very readily to a regulus, with a diminution of weight. Its difficult fuſibility, therefore, muſt have been owing to ſome accidental circumſtance, the effect, perhaps, of the other aſſays. On expoſing the aſſay A. (b) per ſe, to the blaſt, for the ſpace of half an hour, under the ſame arrangement of the forge, its fuſion was not in the leaſt degree advanced. This was tried with ſeveral others of the above-mentioned aſſays, with the very ſame reſult.

D. In order to ſee how far the fuſion might be promoted by ſtill greater additions of copper, the following experiments (with this alteration in the arrangement of the forge, that the diſtance from the mouth of the air-channel or *form* was increaſed to five inches) were made.

(a) 64lbs. copper, and 16lbs. molybdænic calx.

(b) 96lbs. copper, and 16lbs. molybdænic calx.

(c) 80lbs. copper, and 16lbs. molybdænic calx, all laid upon a bed of braſque, with a layer of charcoal duſt, and covered over with a looſe lid.

After expoſure to the blaſt for half an hour, none of the aſſays ſhewed greater tendency to fuſion than (c) which was melted to a round, but flat, button, that was ſcorified in its upper part, and hollow underneath. The aſſay D. (b) was melted to a round form, but ſtill exhibited ſharp upright edges. Being put back again in a covered crucible, without the addition of charcoal powder, it entered into fuſion; but by the agitation which it met with in being taken out of the crucible, it was divided into a number of granules, none of which, however, had a globular form, and between which a good deal of uncombined molybdænic calx lay interpoſed. Theſe granules of copper bore being hammered a little before they ſplit,

ſplit, and being digeſted in nitrous acid, they left undiſſolved a ſmall quantity of greyiſh calx of molybdæna.

E. On adding ſtill larger proportions of copper, ſo as to amount to 90 or 95 in the 100, the fuſion went on little better than before. However, the aſſays made with the laſt of theſe proportions were evidently found the beſt. After a few ſtrokes with a hammer they cracked on the edges, and ſhewed cavities within.

F. As theſe aſſays might perhaps have been too large, trial was made with leſs quantities in ſmaller crucibles. On this occaſion the calx of molybdæna made uſe of was prepared exactly according to Mr. Scheele's directions. It was taken in the proportion of 1, 2, 3lbs. &c. to 15, 14, 13lbs. of copper. The reſults, however, were much the ſame as before. But after ſeveral fuſions upon a bed of braſque, the colour was changed to a yellow white, that pervaded the whole button, which preſented a ſhining ſurface after filing, but ſoon tarniſhed by expoſure to the air. The reſults were the ſame when the

mixture

mixture was in the proportion of 11, 10, and 9lbs. copper, and 5, 6, and 7lbs. calx of molybdæna. In one of theſe aſſays the beginning of a fuſion to a round button was actually obſerved; it was of a yellow white, and ſoon tarniſhed on expoſure to the air.

One ſhould be ready to pronounce that this change of colour was occaſioned by the calx of molybdæna mixing itſelf with the copper, were it not known from other experiments, that by melting copper along with crude molybdæna, a *matt* of a ſimilar colour is obtained. Although it cannot be denied, therefore, that the copper in the above inſtance contained an extraneous admixture or alloy, yet it ought by no means to be aſſerted, that pure molybdænum* conſtituted this alloy. That vitriolic acid is here concerned, is evident from this, that if ſuch copper be expoſed upon charcoal to the blow-pipe, it gives out ſparks of *matt*.

* By *Molybdænum* is meant reduced, or reguline molybdæna.

If

If it be diſſolved in pure nitrous acid, there remains behind a portion of calx of molybdæna that is of a white, and another portion that is of a duſky, of a grey, and of a black colour. Theſe two laſt circumſtances are proofs of a regenerated molybdæna, or of a pure calx of molybdæna combined with hepatic air, which cannot be formed without the preſence of vitriolic acid. During its diſſolution no ſmell of hepatic air was perceived. As to the reſt, the molybdænic metal ſhews in this reſpect the ſame property as other metals, in forming with ſulphur a matt, and afterwards combining in that ſtate with other metals. But that a portion of the molybdænic calx is, in theſe inſtances, actually revived, and, in its metallized ſtate, enters into union with the copper, is rendered probable by the white calciform particles obtained on ſolution. It is not clearly determined by theſe experiments in what proportions the calx of molybdæna and copper unite beſt. Till we come to fuſe the perfect molyb-

dænic

dænic metal itſelf *(molybdænum)* with copper, the proportions in which the two metals moſt readily and moſt intimately combine, cannot be duly aſcertained.

§. XXIX.

Calx of Molybdæna and Platina.

BEFORE he came to know that the calx of molybdæna which he employed contained ſo many extraneous admixtures as he afterwards diſcovered in it, Mr. Hielm performed a number of experiments with it and platina. In order to free the laſt named metal from the particles of iron and other impurities with which it abounds in the ſtate in which we receive it, he digeſted it in ſpirit of ſalt for ſix or eight times ſucceſſively (uſing freſh acid each time) edulcorated the reſiduum, and ſubjected it to a red heat in a cupel. By this treatment it acquired a dark colour, and loſt two or three per cent. in weight.

G. In

G. In theſe experiments the aſſays were mixed and melted in the ſame manner as in the preceding trials, and the calx of molybdæna was of the ſame kind as that employed in the firſt experiments with copper.

(a) 16 lbs. molybdænic calx and 32 lbs. platina.

(b) 16 lbs. molybdænic calx, and 64 lbs. platina.

(c) 16 lbs. molybdænic calx, and 128 lbs. platina.

The firſt and ſecond mixtures were become caked or agglutinated, and acquired an oblong flatted figure; but in reſpect to its bulk, the third was little altered, and was beſides leſs coherent. They were repeatedly expoſed to the ſame degree of heat, without being brought nearer to fuſion. The aſſay (c) was ſo brittle under the hammer as to crumble to a powder. The ſcaly particles of platina, however, had been attacked and diſſolved ſo as to have their former figure deſtroyed. The other aſſays bore ſeveral pretty ſmart ſtrokes of the hammer, before they ſplit, and their ſurfaces were ſome-

ſomewhat ductile. In their fracture they appeared of a bright grèy colour, and of a granular ſteel-like texture. There is no doubt therefore that a ſolution or union took place here; but the quantity of the mixture, and largeneſs of the crucible at the bottom, probably prevented its fuſion to a round button.

H. To obviate this, ſmaller crucibles were made uſe of. In

(a) were put 15lbs. platina, and one lb. molybdænic calx.

(b) 13lbs. platina, and 3lbs. molybdænic calx.

(c) 11lbs. platina, and 5lbs. molybdænic calx.

On taking out the aſſays, (a) was found melted into an oblong maſs or button, that was brittle, white in its fracture, and full of holes; (b) had aſſumed a globular form, but was jagged on the outſide, and ſpungy within; it was alſo very brittle; (c) was in like manner very brittle, but it was perfectly round, had a ſmooth ſurface, and was of a den-

a denſer and more compact texture than any of the others.

From conſidering theſe reſults, Mr. Hielm was led to ſuſpect that the calx of molybdæna might not be wholly free from vitriolic acid; and, though on further examination, he did not detect the preſence of it, yet he perceived, on adding an acid to one of the obtained buttons, an evident hepatic ſmell (In what way this hepatic air is formed during the preparation of the molybdænic calx, with which it unites, the author has before explained) In theſe experiments, therefore, the calx of molybdæna ſeems to have formed with the platina a ſort of matt.

I. It was next tried to melt calx of molybdæna prepared according to Mr. Scheele's directions, with platina; which was effected, though with ſomewhat more difficulty. In this caſe however no hepatic ſmell was diſcoverable.

K. Calx of molybdæna prepared by burning with linſeed oil, was boiled in diſtilled

water

water, the firſt quantity of which was thereby turned of a green colour. This was repeated till the decanted water was quite colourleſs, and the molybdænic earth which remained behind was perfectly white. It was afterwards boiled two or three times more with freſh water, poured out upon a filter of blotting paper, further edulcorated, dried, and ſubjected to a red heat.

It was attempted to unite this calx of molybdæna, by fuſion, with platina. It ſhewed ſome tendency to combine therewith, and the compound thus formed gave out, on the addition of an acid, an hepatic ſmell: a proof, how difficult it is to ſeparate the fixed alkali and vitriolic acid from this calx, which moreover probably contained ſome undecompoſed molybdæna.

L. White, ſhining flowers, obtained from calx of molybdæna prepared with linſeed oil, were, without previous edulcoration, tried to be melted together with platina. They did, in fact, unite therewith, yielding a compound ſimilar to the former in every reſpect.

 Hence

Hence we ſee how obſtinately theſe extraneous ſubſtances adhere to the molybdænic calx, and how carefully they ſhould be ſeparated from it, if we wiſh to obtain proper reſults. As a further illuſtration of this matter, I ſhall here give an account of ſome experiments with iron, which metal affords the readieſt means of diſcovering whether the molybdænic calx contain an admixture of vitriolic acid; for if it do contain ſuch an admixture, the iron becomes red-ſhort.

§. XXX.

Calx of molybdæna with iron.

M. In theſe experiments the cleaneſt iron-filings that could be got from workmen in manufactories, were taken and were further picked and purified before they were uſed. The calx of molybdæna was prepared by treatment with linſeed oil, and ſubſequent roaſting.

(a) 80lbs. iron-filings, and 20lbs. molybdænic calx, gave an oblong button, that was very brittle, of a yellowiſh white colour in its fracture, which exhibited a

fine leafy or ſcaly texture, intermixed with black ſpots. The file made but little impreſſion upon it.

(b) 95lbs. iron-filings, and 5lbs. molybdænic calx. The button from this aſſay was ſo hard as to make indentations in the iron plate on which it was hammered, before it ſplit. In its fracture it exhibited a coarſe, ſcaly texture, a dark grey ſhining colour, with ſome white places on the edges. It yielded ſomewhat to the file, which left many white ſcratches upon it.

(c) 90lbs. iron-filings, and 10lbs. molybdænic calx, gave a regulus which was as hard under the hammer as the laſt, exhibiting in its fracture a lamellar or foliated texture, and a white ground, interſperſed with black ſpots. Like the preceding, it did not ruſt or tarniſh by expoſure to the air for ſeveral weeks. The file made little impreſſion upon it.

All theſe aſſays were well fuſed, and exhibited a ſmooth ſurface, which, however, in every inſtance was globular and prominent above. Pieces of this iron melted readily

before the blow-pipe, with intumeſcence, and with a projection of numerous white and red ſparks, and this, whether the proportion of molybdænic calx was greater or leſs. On the addition of an acid, theſe aſſays emitted a ſmell of hepatic air.

N. Calx of molybdæna purified in the manner above deſcribed at K, was employed in the following fuſions.

(a) 80lbs. iron-filings, and 20lbs. molybdænic calx. The button thus obtained was outwards of the colour of plumbago; it was very brittle, and when broken it ſeemed as if it were compoſed of ſmall cubes, and was of a yellowiſh white, and dark grey, ſhining colour.

(b) 95lbs. iron-filings, and 5lbs. molybdænic calx. This aſſay had externally a dull plumbaginous colour, and in conſequence of the tough coating or cruſt with which it was covered round, it bore ſeveral ſtrokes of the hammer before it broke. Its fracture was uniform, dark grey, and ſhining, interſperſed with white prominent ſurfaces.

(c) 90lbs.

(c) 90lbs. iron-filings, and 10lbs. molybdænic calx. The button from this assay was in every respect similar to the last, but more brittle. They all melted before the blow-pipe, throwing out sparks accompanied with a hissing noise; but these sparks were few in number, and of short continuance. They were not found to yield hepatic air on the addition of acids; but were, like all the rest, attracted by the magnet. These reguli were globular, and convex in their upper part.

O. Instead of filings of forged iron, borings of cast-iron were made use of in the following experiments.

(a) 80lbs. borings of cast-iron, and 20lbs. molybdænic calx, of the same kind as that used in M. yielded a button of a foliated texture, and of a white coloured ground, interspersed with grey dots.

(b) 95lbs. cast-iron-borings, and 5lbs. molybdænic calx. This regulus was fine grained, and of a dark grey shining colour.

(c) 90lbs. caſt-iron-borings, and 10lbs. molybdænic calx. This button had a white ground, dotted with black ſpots.

In other reſpects theſe aſſays reſembled thoſe in M.

A few more experiments relative to the fuſion of iron with the molybdænic calx, remain to be related in this place.

P. 16lbs. of iron-filings, and 16lbs. of a calx of molybdæna that had been ſomewhat edulcorated, gave a regulus which weighed 24lbs. not reckoning ſome ſmall globules which remained inherent in the braſque or lining of the crucible. It was well fuſed, and had a very ſhining ſmooth ſurface. Under the hammer it proved brittle; in its fracture it preſented an uniform granular texture, and a white colour. On the affuſion of an acid, it did not give out any hepatic air; it tarniſhed quickly, and during its fuſion before the blow-pipe, it threw out a great number of ſparks.

(b) 8lbs. iron-filings, and 16lbs. molybdænic calx, prepared after Scheele's man-

ner, melted to a round button, that was very brittle, internally whiter than the preceding, and of a coarſer grain. During its fuſion before the blow-pipe it gave out ſparks, but no hepatic ſmell.

(c) 8lbs. iron-filings and 4lbs. unedulcorated molybdænic calx in flowers, melted to a pretty ſmooth and ſhining button, that weighed 10lbs. was ſufficiently hard and ſolid, of a fine ſteely texture, and dark grey colour; it did not ruſt in the air, but on dropping upon it an acid, a ſmell of hepatic air was perceived. Before the blow-pipe it melted with intumeſcence, during which the molybdænic calx ſeparated from the iron with a hiſſing noiſe, though the iron itſelf emitted but few ſparks.

The changes which the *pure* molybdænic metal is capable of inducing on iron by commixture with it in the fire, remain to be determined by future trials; for in the preceding inſtances the hepar ſulphuris which the molybdænic calx contained muſt be ſuppoſed to have contributed ſomething to the obtained reſults.

 §. XXXI.

§. XXXI.

Calx of molybdæna with regulus of manganese.*

Q. In these experiments Mr. H. melted together

(a) 4lbs. regulus of manganese and 1lb. molybdænic calx, which yielded a round metallic button that was surrounded with a dark-green coloured drossy crust. Internally it was of a dull white colour, differing very little from that of pure regulus of manganese.

(b) 4lbs. regulus of manganese, and half a pound molybdænic calx, yielded an oblong button, which was harder and much whiter than the preceding.

R. Both the preceding assays (Q. (a) (b) were pulverised; they weighed exactly 8lbs.

* For obtaining regulus of manganese, Mr. H. reduces crude manganese (freed from iron and all other heterogeneous matter) to a fine powder, which he makes into a paste with linseed oil, and spreads upon the sides of a crucible properly brasqued with clay and charcoal-powder. In this way he obtains more and larger reguli than he used to obtain when he made the mixture into a single heavy lump, the greater part of which was generally scorified.

and were mixed with an equal weight of calx of molybdæna. The whole was melted to a round metallic button, which was ſurrounded and intermixed with a ſorrel coloured ſcoria; this button, including its ſlag, weighed 14lbs. It melted before the blow-pipe with intumeſcence, and was wholly converted into a ſpongy maſs, part of which was white and part black. Glaſs of borax was tinged by it in the ſame manner as it is wont to be by manganeſe.

§. XXXII.

Calx of molybdæna with gold.

S. Gold of parting, finely pulveriſed was uſed in theſe experiments.

(a) 15lbs. gold powder, and 1lb. molybdænic calx.

(b) 14lbs. gold powder, and 2lbs. molybdænic calx.

(c) 13lbs. gold powder, and 3lbs. molybdænic calx.

(d) 12lbs. gold powder, and 4lbs. molybdænic calx.

Of

Of theſe aſſays the firſt was found in ſome degree melted together, but was very brittle under the hammer, and of a lighter colour than gold itſelf. The other aſſays ſhewed the ſame properties, ſo much the more as the proportion of molybdænic calx that had been added to them, was greater. Even in the above proportions they were inclined to ſpread out into thin plates, rather than to run together into a button. This alſo was found to be the caſe in other trials, in which equal parts of both were taken. They melted into thin, pulverable laminæ, of a very pale yellow colour. Before the blow-pipe the gold ſeparated with a hiſſing noiſe, but without ſparks.

§. XXXIII.

Calx of molybdæna with ſilver.

T. The ſilver here uſed was diſſolved in pure nitrous acid, and precipitated therefrom by copper, and was afterwards edulcorated with warm water and dried.

The

The ſilver powder and molybdænic calx (this laſt was uſed in the form of flowers, which had been obtained by calcining the molybdæna in a capſule or ſaucer, and had been carefully picked out from the reſt of the calx) were mixed together in the ſame proportions as the gold powder. With ſmall additions of the molybdænic calx, ſilver kept its ductility and colour better than gold had done. When equal parts of each were taken, the mixture would not enter into fuſion, but yielded a very porous maſs, of a greyiſh colour and fine-grained in its fracture; whence it may be inferred that theſe ſubſtances had penetrated each other, and that the molybdænic calx here employed was quite pure. If the proportion of calx of molybdæna be conſiderable, the compound does not melt before the blow-pipe, but the ſilver eliquates or ſeparates from the molybdæna. In other reſpects this alloy hiſſes and decrepitates before the blow-pipe in the ſame manner as the alloy with gold. By cupellation with lead the molybdænic calx is readily ſeparated, and gradually

dually confumed. The fame thing happens with the compound of gold and molybdæna. On the addition of nitrous acid the filver is diffolved, and there remains behind white calx of molybdæna, with a little that is of a dufky colour.

†*† The *lifpound* fo frequently mentioned in Mr. Hielm's experiments, contains 14 Mecklenburgh, 15 Hanoverian, and 16 Dantzic pounds. We have all along rated it at 14 lbs.

(To be concluded in the Appendix.)

XXIV. EXPERIMENTS ON, AND ANALYSIS OF The MAGNETIC SAND,

Found in the County of CORNWALL, and called by Mr. GREGOR, *MENAKANITE.*

By Mr. SCHMEISSER*.

THIS fubftance is found in the form of black grains, of the fize of gunpowder, but varioufly fhaped.

* Communicated by the Author.

When

When ſeparated from the grey ſand with which it is generally mixed, I found its ſpecific gravity to that of diſtilled water, as 4,800 : 1,000.

It is eaſily reduced to fine powder, and is ſtrongly attracted by the magnet.

1ſt. Having kept it four hours in a red heat, I found its weight increaſed two per cent. and its colours turned much lighter.

2ndly. Boiling water takes up a ſmall quantity, and thereby acquires a milky appearance. It paſſes of the ſame colour through filtering paper; paper ſtained with litmus or turmeric is not affected by this mixture.

3rdly. When mixed with nitre and thrown into a red hot crucible, it does not detonate.

4thly. Volatile fluid alkali when poured on it, does not produce any blue colour.

5thly. Mixed with borax and expoſed to the blow pipe, the borax acts in ſome degree on it, ſeems to diſſolve part, and extracts a little greeniſh colour, which afterwards changes to a browniſh hue.

6thly. It

6thly. It is ſcarcely acted upon in its granulated ſtate by microcoſmic ſalt, but is ſomewhat more acted upon when previouſly reduced to powder; the flux at firſt appears of a greeniſh colour, which diſappears nearly on cooling, and becomes browniſh; it does not efferveſce with any acid.

7thly. When melted with fixed alkali, the maſs appears after cooling of a reddiſh brown colour. On adding a little pure nitrous acid to the filtered ſolution of this compound in diſtilled water, a trifling precipitation takes place, ſimilar to that which is obtained on adding nitrous acid to a very much diluted liquor ſilicum.

8thly. When aqua regia is poured on the powdered menakanite, a great part of it is diſſolved, and the ſolution appears green. On mixing this with a ſolution of pure tin, and alſo of vitriol of iron, no blue colour is produced.

9thly. The marine acid diſſolves a large proportion of this ſubſtance powdered, but does not appear to be in any degree dephlogiſticated by it.

10thly. By

10thly. By digeſtion in nitrous acid, but little is diſſolved. Marine acid added to this ſolution, does not throw down any precipitate.

11thly. Vitriolic acid acts the moſt upon this ſubſtance, and being ſaturated with it, leaves a white powder behind.

12thly. No dephlogiſticated air is produced on expoſing the powdered menakanite to a ſufficient degree of heat.

13thly. Placed in a crucible with charcoal, and covered with borax and powdered glaſs, and expoſed to a proper degree of heat, a regulus is produced, which is attracted by the magnet; the regulus diſſolved in vitriolic acid, yielded on evaporation cryſtals ſimilar in ſhape and colour to thoſe of vitriol of iron.

Two hundred grains of this ſubſtance, reduced to a very fine powder in a glaſs mortar, were mixed with 600 grains of dry mineral alkali, and well calcined in a crucible. The maſs when cold was of a reddiſh brown colour; this was moiſtened with diſtilled water, and ſaturated with aqua regia;

gia; the ſaturated ſolution was filtered, and the remainder again extracted with a ſufficient quantity of aqua regis; the reſiduum after being edulcorated with diſtilled water and thoroughly dried, weighed 180 grains, and was ſtill of a browniſh colour.

Theſe 180 grains of reſiduum were again mixed with 200 grains of mineral alkali, calcined as before, and ſaturated with aqua regia.

This reſiduum being further extracted with aqua regia, edulcorated and dried, weighed 156 grains. Theſe were again mixed with alkali, and ſubmitted to the ſame operation, which was repeated until the aqua regia would not extract any more; when 64 grains ultimately remained. 20 grains of this white powder were mixed with 60 grains of vegetable alkali, and properly calcined.

The maſs after cooling, was lixiviated with diſtilled water, and ſuperſaturated with nitrous acid. A ſmall quantity of a nitrated ſolution of ponderous earth was added, but no ponderous ſpar was produced. The white powder,

powder, when expoſed to a very ſtrong heat per ſe does not undergo any alteration.

Ten grains of this white powder were mixed with 40 grains of vegetable alkali, and expoſed to a heat ſufficient for melting glaſs; the maſs when cold deliqueſced in the air, and the white powder was again left ſeparate. After being edulcorated, dried, and carefully collected, it was found to have loſt only one grain.

Twenty grains of the white powder were digeſted with fluor acid. The acid diſſolved two grains, which were found to be calcareous earth.

Some of the white powder was expoſed to the blow pipe with microcoſmic ſalt, but was not diſſolved.

A microſcope ſhewed the white powder to be ſuſpended in a glaſs button, which had been formed.

Borax ſeems to act more upon this powder, and produces a milky and purple coloured glaſs, which diſſolves in the vitriolic acid, and when precipitated again with

aërated alkali, it becomes ſoluble in marine acid and water, but not in the nitrous.

No perfect glaſs could be produced by expoſing it to a melting heat with fixed alkali, conſequently it muſt be different from the earth which has hitherto been called ſiliceous earth, and different from the other earths, being only ſoluble in vitriolic acid when in its firſt ſtate.

Mr. Gregor has obſerved, that it prevents iron from precipitating of a blue colour when mixed with phlogiſticated alkali, and alſo from producing ink with an infuſion of galls.

The whole of the extractions collected from experiments 13, 14, 15, were afterwards nearly ſaturated with pure mineral alkali, and divided into two equal parts. Into one portion was gradually dropped ſome phlogiſticated alkali, when a fine blue coloured precipitate was produced, which kept its colour for a conſiderable time; after due calcination it weighed 88 grains, and was wholly attracted by the magnet. After deducting

ducting the quantity of iron contained in the employed phlogisticated alkali, there remained 67 grains, as the proportion of iron calx contained in half the solution.

The other portion of the solution was precipitated with caustic volatile alkali; the obtained precipitate, after being edulcorated, dried, and calcined, weighed 66 grains, and was all taken up by the magnet. Volatile alkali poured on it, did not extract any blue colour.

The liquors which remained after both the above precipitations were mixed together, and completely saturated with aërated alkali, when three grains of an earth were separated, which proved to be argillaceous earth, yielding alum with vitriolic acid.

200 grains therefore of this magnetic sand, consist of

Iron calx - - -	132 grains
Silex - -	$6\frac{1}{2}$
Argill - - - -	3
Menakanite substance or earth	58

XXV. EXPERIMENTS AND OBSERVATIONS ON MANGANESE;

By PROFESSOR FUCHS.

IT is now ſome time ſince Mr. Bindheim publiſhed an account of his ſucceſsful attempts to obtain regulus of manganeſe in the humid way*. For this purpoſe he diſſolved the aërated calx of manganeſe in nitrous acid, and ſet the ſolution over the open fire, in a glaſs retort. During the abſtraction of the acid, the manganeſe, in a ſhining metallic form, depoſited itſelf on the ſides of the retort. After I had printed off my Hiſtory of Manganeſe, I had opportunity of trying Mr. Bindheim's method; and ſhall here relate, in few words, the mode in which I proceeded, together with the reſults.

* See the ſecond Volume of this Journal, page 46.

I diſſolved

I diſſolved $7\frac{1}{2}$ drachms of vitriol of manganeſe in a ſufficient quantity of water, and gradually precipitated the calx with half a pound of ſalt of tartar diſſolved in water. The obtained precipitate, after edulcoration and drying, was of a white colour, and weighed 2 drachms 10 grains. This quantity of precipitate was put into a glaſs retort, 2 ounces of nitrous acid were poured upon it, the retort was ſet in the ſand bath of a ſmall caſt-iron furnace, and a receiver was fixed to it. At firſt there came over colourleſs nitrous acid, which after 7 hours appeared in deep red or orange coloured vapours, like ſmoking ſpirits of nitre. When the whole was become cold, I found the retort ſtill full of red vapours; but its inſide was covered over or lined with a metallic depoſit, which was ſhining, but porous; it was very friable, and weighed 2 drachms. Underneath a few white ſpots were obſerved. Some particles of the depoſit could not be ſcraped off from the ſides of the retort, which accounts for the 10 grains that were miſſing.

Mr. Weſtrumb and Dr. Hermbſtadt have already remarked, that oil of vitriol becomes ſo much altered by manganeſe, as to ſhow properties quite different from thoſe which it poſſeſſed before. I accidentally obſerved in oil of vitriol, which had ſtood for ſome length of time over manganeſe, ſome ſingular properties which I never heard of before, and which I ſhall here mention. About Chriſtmas, 1790, I poured upon half an ounce of manganeſe one ounce of oil of vitriol, and let them ſtand together for at leaſt half a year. Some time after, wanting ſome oil of vitriol for the preparation of vitriol of zinc, I employed for this purpoſe half an ounce of that above-mentioned, diluted with 2 ounces of water ; but no heat enſued, as happens with the common oil of vitriol, notwithſtanding that it gave out fumes, and yielded a good vitriol of zinc. I was ſtill more ſurpriſed to find, on the 5th of February, 1791 (when there was a heavy fall of ſnow) that the other half ounce, which ſtill remained ſtanding over the manganeſe, and had acquired a yellow colour,

was cryſtallized in the ſame manner as we ſee glacial oil of vitriol to be. The cryſtals were radiated, and diſſolved, and re-appeared according as the cold was greater or leſs; thus, on the 22d and 23d of March, there was a froſt during the night, and the oil of vitriol was become cryſtallized; on the 24th there appeared a ſmall quantity of yellow liquor over the cryſtals; but this liquor was congealed again during the four next days: This congelation continued, with ſlight liquefactions every now and then, for the ſucceeding days, till the 7th of April, when the whole was again become fluid. The bottle ſtood at the window in a very cold room. In order to ſee whether the cryſtallization of the oil of vitriol depended on the manganeſe, I put an ounce of freſh oil of vitriol into a bottle without manganeſe, as early as the month of February; but it did not congeal. Hence may we not ſuppoſe that it is owing to the great quantity of phlogiſton which it contains (and of which in the inſtance above

 related,

related, it is deprived by the manganeſe) that common oil of vitriol will not congeal in a moderate degree of cold?

XXVI. SOME REMARKS

CONCERNING THE

ACID OF ARSENIC;

By Mr. WIEGLEB.

(An Extract.)

IN Scheele's Treatiſe on Arſenic, are deſcribed two methods of decompounding and dephlogiſticating arſenic, and obtaining its acid. In this treatiſe we are told, among many other things, that the acid which remains behind (in either of the

the proceſſes) weighs nearly as much as the arſenic employed*. On this point Sir Torbern Bergman has ſpoken in more definite terms, aſſerting, that 100 parts of white arſenic leave after dephlogiſtication about 80 parts of dry arſenical acid. Hence he infers, that the loſs of weight is to be aſcribed to the inflammable principle which is diſengaged, and whoſe weight amounts to 20 in the hundred†.

In the year 1783, having obtained from 8 drachms of white arſenic (treated according to Scheele's firſt method) 9 drachms of arſenical acid, Mr. W. was ſurpriſed at a reſult ſo contradictory to the before-mentioned ſtatements of Scheele and Bergmann, and began to doubt the juſtneſs of the former author's theory reſpecting this acid, and to ſuſpect, that, notwithſtanding the heat that was applied, a portion of the acid diſ-

* Scheele's Eſſays (Engliſh tranſlation) page 146.

† Bergman Opuſc. phyſ. chem. Vol. II. p. 294 (Engliſh Tranſlation, Vol. II. p. 291, 292.)

tilled therewith might remain combined with the arſenic, and that the acid nature of the reſiduum might be entirely owing to this. Not being able, however, to bring this matter to a demonſtration, he let the ſubject alone, in expectation that it might perhaps be cleared up on ſome future occaſion.

The ſyſtem of Lavoiſier having lately engaged his attention, Mr. W. ſet about repeating all the phlogiſtic proceſſes with which he was acquainted, and amongſt the reſt he bethought himſelf of his former obſervation reſpecting the arſenical acid. "I reduced (ſays Mr. Wiegleb) 4 drachms of white arſenic to powder, which I put into a ſmall glaſs retort (the retort was previouſly weighed) poured upon it 14 drachms of rectified white marine acid, and ſet the mixture to diſſolve in a ſand bath under the application of a boiling heat. As ſoon as I could take the retort, after letting it cool a little, out of the ſand, I poured into it 7 drachms of nitrous acid, whoſe ſpecific gravity was 1,200, and diſtilled the acid to dryneſs

nefs with the requifite degree of heat. Here an appearance occurred which prevented me from continuing the fire till the refiduum became red hot. Towards the end of the diftillation, while the contents of the retort were yet perfectly fluid, there attached itfelf to the upper part of the neck of the receiver, a tranfparent cryftalline fublimate, part of which was gradually diffolved by the warm vapours, and trickled down into the acid in the receiver. When all the acid was abftracted, there remained at the bottom of the retort, a folid white cake. On weighing the retort with it, I found, after deducting the weight of the empty retort, that the refiduum amounted to 3 drachms, 2 fcruples."

"From what happened in this experiment, I concluded that the quantity of nitrous acid which had been employed, was not fufficient for the complete dephlogiftication of the arfenic, and hence that a portion of the undecompofed mineral had been forced up in the form of vapours. I therefore collected all

all the ſublimate in the receiver, and poured it back again, along with the diſtilled liquor, into the retort, adding thereto an ounce of freſh nitrous acid. The whole of the acid with ſome red vapours, but without the leaſt appearance of ſublimate, now come over, and the reſiduum was heated till it became red hot. On taking the retort out of the ſand after it was become quite cold, and weighing it together with the white cake of arſenical acid which it contained, I found, after deducting the weight of the empty retort, that the arſenical acid now weighed four drachms and a half."

"Thus it is ſeen, that in the diſtillation of nitrous acid over arſenic, a portion of this laſt is capable of being ſublimed and carried over with the acid, if the dephlogiſtication be not complete. This probably happened in Scheele's and Bergmann's proceſſes; with the former in a ſlighter degree than with the latter. It further follows from theſe experiments, that white arſenic by no means loſes, as

Bergmann has aſſerted, 20 per cent. by dephlogiſtication (for this loſs which he experienced was owing to the ſublimation of a portion of the arſenic) but, on the contrary, that the reſiduous arſenical acid acquires an increaſe of about 12 and a half per cent in weight*."

* Mr. Weigleb accounts for this augmentation of weight on the ſuppoſition that the inflammable principle diminiſhes the natural or original gravity (*urſprungliche ſchwere*) of all ſubſtances with which it is combined. According to this hypotheſis, arſenic muſt be rendered heavier by the abſtraction of this principle. This aſſumption, and the explanation drawn from it, few philoſophers, we preſume (chemiſts or not chemiſts) will be diſpoſed to admit.
EDITOR.

XXVI. ON

XXVII. ON THE POWER WHICH CHARCOAL POSSESSES OF RENDERING *Coloured Liquors colourless*, AND OF CORRECTING AND DESTROYING THE FETOR PRODUCED BY PUTREFACTION;

By Mr. KELS*.

(An Extract)

PART I.

§. I—II.

FOR depriving two ounces of common syrup, diluted with 16 ounces of water, of its brown colour, 12 ounces of charcoal powder were found necessary. The charcoal

* It were to be wished that some of our own chemists would repeat these and Mr. Lowitz's experiments on the clarifying and antiseptic properties of charcoal,—properties which promise to be applicable to a variety of purposes in œconomy, pharmacy, and the arts. EDITOR.

coal was thrown in at 3 different times (four ounces each time) and the boiling was continued at each addition of the powder, for a quarter of an hour. Mr. K. attributes Mr. Gottling's *, and Dr. Hahneman's † failures in their attempts to purify common ſyrup, to the employment of too ſmall a quantity of charcoal.

§. III.

By diſtilling eight ounces of common ardent ſpirits (corn or malt ſpirits) over one ounce of charcoal powder, the ſmell and taſte were conſiderably improved.

§. IV.

By ſhaking eight ounces of the ſame ſort of ſpirits for ſome minutes, with one ounce of charcoal powder, and afterwards ſeparating the charcoal by filtration, the ſmell and taſte of the ſpirits were ſomewhat improved. Mr. K. however acknowledges that he has found, that not only by agitation (as Dr.

* Pocket-book or Almanack for Chemiſts for 1790, p. 38.
† Crell's Annals for 1789, Vol. I. p. 202.

Hahne-

Hahneman* firſt remarked) but alſo by diſtillation with charcoal powder, the ſpirit contracts a pungent or biting taſte.

§. V.

Spirit of wine that had been diſtilled with carraway ſeeds was, as Mr. Lowitz had before aſſerted, deprived of the carraway ſeed ſmell, by repeated diſtillation with charcoal powder. The ſame effect was alſo produced by agitation with a large proportion of charcoal. It was moreover found that ardent ſpirits impregnated with various other aromatic ſubſtances might be in a great meaſure deprived of their acquired odour, by the ſame means.

§. VI.

In order to determine further the power which charcoal poſſeſſes of deſtroying odours, half a dram of oil of lavender, of an exquiſite fragrance, was mixed with two drams of charcoal powder, and diſtilled in a retort, provided with a receiver con-

* Crell's Annals for 1789, Vol. I. page 204.

taining a little water. A gentle heat was at firſt applied, and the fire was kept up till there ceaſed to appear any more vapours in the neck of the retort. On examining the receiver, the diſtilled oil was ſtill found to poſſeſs its peculiar odour, but not (as Mr. K. thought) in ſo high a degree as before the diſtillation.

§. VII.

One grain of muſk was triturated into a a thin paſte with an ounce of charcoal powder and water; but neither at firſt nor afterwards did the muſk-ſmell ſeem to be weakened. With a mixture of two grains of aſafœtida and one ounce of charcoal powder, the reſult was the ſame.

§. VIII.

Water impregnated with hepatic air is not deprived of its fetor by charcoal powder. Mr. K. thinks it probable that charcoal in a humid ſtate is not capable of abſorbing air, as it is, according to Fontana's

and Sennebier's experiments*, in a red hot, or even in a dry ſtate.

§. IX.

One ounce of charcoal powder was mixed with ſix ounces of water, to which was added one ounce of expreſſed oil of rape-ſeed (muſtard ſeed ?) of a dark colour and bad ſmell; the mixture was ſhaken for ſome minutes, and ſet by in a warm place for a couple of days; after which it was attempted to ſeparate the oil from the charcoal powder, an operation which was attended with great difficulty, the greater part of the oil being united with the charcoal. That which could be expreſſed from it, was of a black colour in conſequence of the charcoal that was ſtill mixed with it. It was therefore filtered through paper, by which means a ſeparation was effected; but in this way it was not poſſible to collect more than two drams of oil, which was perfectly colourleſs, like white olive oil, had entirely loſt

* Annales de Chimie, Tom. 4, p. 261.

its bad ſmell, and was well taſted; though Mr. K. thought that this rectified oil was diſpoſed to become rancid more quickly than the ordinary oil.

On account of the great abſorption of oil by the charcoal powder, and the difficulty and loſs attending the ſubſequent ſeparation, it is not much to be expected (as Mr. Gottling has already remarked*) that charcoal will be applied to the purification of expreſſed oils, in the great.

§. X.

The aſtringent principle in vegetables is deſtroyed by charcoal. To produce this effect, an enormous quantity of charcoal powder is requiſite. Infuſion of galls was boiled repeatedly and gently with charcoal powder, and afterwards, without being made warm, was ſhaken for ſome time with the ſame, and then left at reſt. On account of the great quantity of charcoal powder that was employed, it was neceſſary to dilute the

* Almanack for Chemiſts for 1791, p. 81.

infuſion largely, but it was afterwards reduced to its former quantity by evaporation. Freſh infuſion of galls acquired from the charcoal at firſt a dark blackiſh-green colour, which, however, was entirely taken away by ſubſequent agitation with more charcoal and by filtration through the ſame. Infuſion of galls that had ſtood by for ſome time, did not exhibit this blackneſs. Both became at length colourleſs, betrayed no aſtringency to the taſte, produced not the leaſt blackneſs in a ſolution of vitriol of iron, precipitated nitrated quickſilver white, did not redden paper ſtained with litmus, but rather made it of a deeper blue colour. May not the deſtruction of the aſtringent principle be owing to the particles of iron, which, as appears from Mr. Weſtrumb's experiments charcoal contains?

§. XI.

It had been before obſerved by Mr. Lowitz, that water coloured by litmus was deprived of its colour by charcoal. This induced Mr. K. to try its effects upon other pigments.

pigments. Accordingly, he took four ounces of water coloured of a deep blue by a ſolution of indigo in vitriolic acid, and added thereto half an ounce of charcoal powder: The mixture, after agitation for a few moments, paſſed through the filtre as colourleſs as water. In like manner watery tinctures, or infuſions of cochineal, fernambuck, and madder, were rendered colourleſs by charcoal powder.

§. XII.

For the purification of crude tartar, both red and white, charcoal powder was found very uſeful. Two drachms of red tartar were boiled for a quarter of an hour with half an ounce of charcoal powder and eight ounces of water; the ſolution was colourleſs, and yielded by evaporation a very good white tartar. Mr. K. is therefore of opinion, that charcoal powder may be advantageouſly employed for this purpoſe, in the great. By the ſame means he has frequently prepared from crude tartar, cryſtals of tartar

as white as thoſe which are obtainable from purified tartar (cream of tartar.)

§. XIII.

In repeated experiments he has experienced the efficacy of charcoal in the preparation of a white terra foliata tartari, or acetated vegetable alkali. The failures of other chemiſts in this reſpect he ſuppoſes to have been owing to the employment of too ſcanty a proportion of charcoal powder.

§. XIV.—XV.

By gentle boiling for a ſhort time with charcoal powder, ſoluble tartar (alkali vegetabile tartariſatum) obtained from crude tartar, may be freed from its brown colour, and be brought to a degree of purity equal to that which is prepared from cream of tartar. In the ſame manner the ſoluble tartar which is collected in the preparation of the eſſential acid of tartar, and which for the moſt part is coloured, may be brought to perfect whiteneſs.

PART II.

ONE of the moſt remarkable properties of charcoal, which was firſt made known by Mr. Lowitz, is its deſtroying the ſtench which ariſes from putrid fleſh; for on kneading the ſame with charcoal powder, the fetor is entirely taken off, and nothing but a pure ſmell of volatile alkali remains. This fact has been ſince confirmed by Mr. Brugnatelli*, and alſo by Mr. Kels.

But charcoal by no means checks the putrefactive proceſs which the fleſh is undergoing; for in ſpite of admixture therewith, the fleſh becomes at length completely reſolved or deſtroyed. It is not, therefore, an antiſeptic in the ſtrict ſenſe of the word, ſince it only acts upon the fetid effluvia from the putrefying ſubſtances.

This property of charcoal ſuggeſted to Mr. K. the correction of foul and ſtinking water by the ſame means. His firſt experi-

* Crell's Annals for 1789, vol. 2. p. 50.

ments on this ſubject, which he made in January, 1789 (the reſults of which he communicated to Profeſſors Gmelin, Blumenbach, and Feder, of Gottingen) ſucceeded agreeably to his expectations and wiſhes. It was not (we are told) till after his experiments had been made known, that Mr. Lowitz publiſhed his method of purifying foul water by the ſame means, only with the addition of vitriolic acid. (See the 1ſt vol. of our Journal, p. 202.)

The correction or reſtoration of putrid water by means of charcoal is ſo eaſy, ſo ſimple, and ſo cheap a proceſs, that Mr. K. ſtrongly recommends it to be adopted (in preference to all other methods hitherto propoſed*) in ſea-voyages. In all his ex-

* Namely, ventilation, recommended by Foſter;—ſhaking the water in the caſks after opening them, recommended by Aſkow and Lind; who further recommends boiling the putrid water briſkly but for a ſhort time, and alſo the addition of alum;—the addition of quicklime, recommended by Foſter (Mr. Kels has found that this laſt, as is noticed in the text, may be uſefully employed in conjunction with charcoal);—nitrated ſilver. This, which has been recommended as a powerful antiſeptic by Dr. Hahneman, was tried by Mr. Kels without effect. It is, beſides, an unſafe article;—laſtly, argillaceous earth or clay, recommended by Mr. Beckman.

periments

periments he has found charcoal powder, added in a greater or leſs proportion; according to the degree of putrefaction and the quantity of putrid particles, to render foul water ſweet by agitation therewith for a few minutes. The ſubſequent ſeparation of the charcoal powder from the water is effected with little difficulty, even in the great.

The employment of quicklime in conjunction with charcoal, was found to contribute conſiderably to the purification of the foul water, eſpecially if the water abound with extractive matter, which the quicklime precipitates, and thereby fines the water and renders it perfectly clear. But he never could take off the bad ſmell of the water by lime alone.

The purifying operation of quicklime, in conjunction with charcoal, is moſt ſtriking in the caſe of water in which flax has been ſuffered to putrefy; ſuch water has a peculiar, and highly offenſive ſmell. That which was uſed in theſe experiments was as black as ink; by addition of quicklime it was ſoon rendered quite clear and limpid,

the

the lime carrying down with it black flakes to the bottom of the veſſel; but the bad ſmell was ſo far from being deſtroyed or removed, that it was on the contrary made worſe, and could only be gotten rid of by the addition of charcoal powder. Water in which cabbage had been ſteeped, and which was of a yellowiſh brown colour, was in like manner rendered fine and clear by quicklime, but did not part with its ſtinking ſmell, till charcoal was mixed with it.

For the ſeparation or precipitation of the portion of lime which in this mode of purification remains diſſolved in the water, Mr. K. propoſes (in preference to fixed air) the employment either of acid of tartar, or of vitriolic acid.

Water that has undergone putrefaction, is deprived of its freſh and briſk taſte, which charcoal is not capable of reſtoring; hence the water that has been thus purified, taſtes ſoft and vapid. For the reſtoration of its pleaſant refreſhing taſte, Mr. K. recommends the employment of fixed air.

The

The disagreeable mouldy taste which some water has in its natural state, may be corrected by simple filtration through charcoal powder; which has this further advantage, that it separates at the same time, the extraneous matter that is mechanically mixed with the water, and thereby renders it pure and clear.

Mr. K. is induced to think that charcoal is even capable of preserving water from becoming putrid for a long space of time; for he could not perceive the least putrid smell in water that had been kept, mixed with charcoal, for a whole year. He accounts for this effect by supposing that the charcoal does not, like a true antiseptic, keep off the putrefactive process, but only absorbs the putrid particles immediately as they are formed, and so prevents them from acting as a ferment to the rest of the water. He therefore thinks it worth while that this experiment be tried on large quantities of water in sea-voyages.

PART III.

AS charcoal has been found to take off the fetor from putrid fleſh, Mr. K. thinks it may be advantageouſly employed on ſhipboard, for ſweetening ſtinking meat. For this purpoſe, the meat needs only to be rubbed in, and remain covered over with, charcoal powder, for ſome time, and afterwards waſhed clean.—He further thinks that it might be uſeful to add ſome charcoal powder at the time the meat is ſalted.

The bilge-water, which ſo much contaminates the whole air in ſhips, might alſo be corrected by means of charcoal powder.

Dr. Crell has propoſed to correct, by the ſame means, the offenſive exhalations in neceſſaries; and Mr. K. thinks that it might be turned to the ſame uſe in hoſpitals.

Charcoal powder is alſo recommended as a medicine. Mr. Monch has lately adminiſtered it with good effect in a caſe of putrid bilious fever (ſynochus bilioſo putrida)

da) in the doſe of half a drachm ſix times in the day, employing at the ſame time, evacuating medicines. He alſo found that by ſprinkling it upon old ulcers, the fetid ſmell of the pus was taken off.

Mr. Kels has obſerved the ſame thing with regard to ulcers; and he thinks that charcoal powder may be given with advantage in all caſes in which the primæ viæ are loaded with putrid and offenſive matter.

He has ſeen charcoal powder mixed with water, employed with good effect, as a waſh for the mouth, to correct a ſtinking breath, which was probably owing to a rotten tooth: hence he infers that the intolerable fetor which ariſes from carious bones, and ulcerated cancers *, might in like manner be removed by charcoal powder.

* And alſo from mortifications?

In Mr. Gadolin's paper (inſerted in the 2d. Vol. of this Journal) on the purification of ſalt-petre by charcoal powder, for "*three-quarters of an ounce*" (p. 201—202—205—207) of charcoal powder, read "*one ounce and a quarter*."

XXVII.

XXVIII. *CHEMICAL NEWS.*

AS Mr. Weſtring's experiments on the lichens will probably attract the notice of ingenious dyers in this country, we ſhall here inſert (from Crell's firſt number for 1792) ſome further particulars relative to this ſubject which have not been mentioned in the Annales de Chimie, from which the account that appeared in our laſt number was taken, before the German Annals had reached our hands.

1°. The lichens (which ſhould be gathered after rainy weather, as they are then eaſily detached from the ſubſtances to which they adhere) muſt be well picked, cleaned and waſhed, and afterwards dried and reduced to fine powder. 2°. In the macerations, digeſtions, and decoctions, river-water ſhould be made uſe of; and by no means ſpring-water, as it ſpoils the colours. To every half ounce of powdered lichen ſhould be taken 12 ounces and a half of water. 3°. The freſher the quicklime, the better; it ſhould not be employed in a greater

greater proportion than one-tenth part*, otherwiſe the articles that are dyed, will be liable to be corroded and made rotten. 4°. The ſal ammoniac is to be added in the proportion of one-twentieth part only; ſo little of it ſuffices. 5°. Woollen yarn ſhould be previouſly boiled with fine bran or pollard, which is all the preparation that it requires. Silk that has been cleanſed and not ſulphured, requires no preparation before dying. Raw ſilk muſt be made clean and white with ſoap. The ſtuffs or articles that are to be dyed muſt be plunged into cold water both before they are thrown into, and after they are taken out of the dying liquor or bath. 6°. When lime and ſal ammoniac are uſed, the veſſel ſhould be covered during the two firſt days. 7°. When the dying liquor or bath is ſtrong, and the quantity of articles dipped in it proportionably too little, the firſt ſamples are not ſo fine as the ſecond. 8°. In this way may be dyed wool, ſilk, camel-hair (whoſe peculiar luſtre adds to the beauty of the co-

* In the extract from the Annales de Chimie in our laſt number, p. 188, for *ten parts* read *a tenth part*.

lours

lours that are given to it) horſe-hair, and feathers. This proceſs for dying with the lichens does not take up more than five or ſix days; whereas, if urine (according to the old method) were to be employed inſtead of quicklime and ſal ammoniac, the operation would require full as many weeks.

Mr. Weſtrumb has confirmed by repeated experiments what Profeſſor Gren had before aſſerted, viz. that in the reduction of mercurius præcipitatus per ſe, by heat alone, no dephlogiſticated air is let looſe.—Half an ounce of mercurius calcinatus per ſe, or hydrargyrum per ſe oxydatum, that had been prepared by Mr. W. himſelf, was put into a ſmall retort, the neck of which was three feet long. To this neck was luted a tube which was inſerted into a double mouthed bottle or receiver, by means of which it was connected with a pneumatic apparatus. The junctures were luted with plaſter of Paris, ſpread upon coarſe linen rag, over which were wrapped ſtrips of linen cloth, ſpread over with a luting made of lime and cheeſe. The retort was ſet in a crucible, and after being ſurrounded with ſand, it

was

was expoſed to the heat of a good wind-furnace. Scarce had the crucible become red hot when there appeared in the neck of the retort clear drops of water, which gradually increaſed and collected in the bottle or receiver. Theſe drops of water were followed by quickſilver in a revived or running ſtate, but not a ſingle bubble of air was obtained. Mr. W. repeated theſe experiments, employing calx of quickſilver and phoſphorus, calx of quickſilver and ſulphur; and the reſults in all his trials were contradictory to the antiphlogiſtic ſyſtem.

From theſe experiments Profeſſor Gren concludes, that all thoſe chemiſts who have obtained dephlogiſticated air in the reduction of calx of quickſilver by heat alone, have employed not the true mercurius præcipitatus per ſe, but the common red precipitate of mercury, prepared with nitrous acid. He aſſerts, that it is from the particles of nitrous acid which adhere to the red precipitate ſo prepared, that the dephlogiſticated air is derived, and that none can be extracted from the pure calx itſelf. He intends to repeat the experiment with æthiops

per ſe, or quickſilver reduced to a calciform ſtate by mere agitation.

The 2d part of the Phil. Tranſactions for this year, contains the following papers on chemical ſubjects; viz. "Further obſervations on the proceſs for converting caſt into malleable iron; by Dr. Beddoes," who here proves (what he had advanced in a former paper on this ſubject, for which ſee the 2d vol. of this Journal, p. 227) that caſt iron, during its converſion into a malleable ſtate, parts with an elaſtic fluid, which conſiſts of inflammable air, ſometimes mixed with carbonic acid or fixed air.—An hepatic ſmell was alſo perceived in the extricated air.—The author thinks, with great reaſon, that his obſervations may ſerve to direct attempts to render the metallurgy of iron leſs difficult, laborious, and expenſive.—"Experiments made with the view of decompounding fixed air or carbonic acid; by Dr. Pearſon." Theſe experiments were ſuggeſted by thoſe which were made by Mr. Tennant (ſee the 1ſt vol. of this Journal, p. 205) who, finding that charcoal and phoſphoric acid were produced by applying phoſphorus

phosphorus to red hot marble, inferred that the carbonic acid, or fixed air of the marble, was decompounded. By applying phosphorus, under a due degree of heat, to mild or aërated fossil alkali, vegetable alkali, calcareous earth, barytes, magnesia, and clay, Dr. P. has obtained similar results, viz. respirable air and charcoal, with phosphoric acid from the union of the phosphorus with the respirable air.—" On the cause of the additional weight which metals acquire by being calcined; by Dr. Fordyce." Pure zinc was dissolved in diluted vitriolic acid, and precipitated from this solution by caustic fixed alkali. The calx thus obtained weighs heavier than the quantity of metal employed. This increase of weight must be owing to matter added to the calx either from the acid, the alkali, the water used in the solution, the air lying on the surface of the materials during the operation, or it must come through the vessels in which the operation is performed. By experiments simple, but ingeniously contrived, Dr. F. shows that the additional weight of the calx, above that of the metal, is not derived from the acid, nor from

from the alkali, nor from any ſubſtance paſſing through the glaſs veſſels, nor from the ſuper-incumbent air during the precipitation, *but from the water.*

[In No. I. of this vol. p. 62, line 3, for *bottle*, read *pint bottle*.]

XXIX. LIST OF

NEW CHEMICAL PUBLICATIONS.

AN account of experiments to determine the ſpecific gravities of fluids, thereby to obtain the ſtrength of ſpirituous liquors. By J. Ramſden. 8vo. 1792.

A treatiſe on the mineral waters of Harrowgate. By Thomas Garnett, M. D. 8vo. 1792.

Pharmacopœia Coll. Reg. Med. Edinburg. 8vo. 1792.

Plan of a courſe of lectures on mineralogy. Tranſlated from the German of Profeſſor Werner, by John Hailſtone, Woodwardian Profeſſor in the Univerſity of Cambridge. 8vo. 1792.

On the properties of matter, the principles of chemiſtry, &c. By E. Peart, M. D. 8vo. 1792.

XXX. FURTHER EXPERIMENTS

ON

W O L F R A M;

By PROFESSOR GMELIN.

PARTLY with a view of aſcertaining whether it contains a peculiar metal, and partly in order to ſee what effects it is capable of producing upon other metals when fuſed therewith, I reſumed * my experiments on Wolfram, with a ſupply of which, from the Bohemian mines, near Slackenwald, Baron Born was ſo obliging as to furniſh me. It is true, that the combination of this ſubſtance with other metals has been already effected by Meſſrs. d'Elhuyarts; but in their experiments the calx of Wolfram was employed alone. Now it ſtruck me that the reſult would be different if this calx were mixed with charcoal pow-

* See Nos. II. and III. of this Volume, p. 127,—205.

der (ſomewhat after the manner that is practiced in the making of braſs with calamine) and expoſed to the fire, along with the other metal, in a crucible lined with charcoal duſt.

Accordingly, I freed ſome Wolfram as completely as poſſible from the quartz through which it was interſperſed, triturated it to a fine powder, and paſſed it through a hair ſieve. One pound of this was mixed with two pounds of levigated potaſh, and ſubjected to the fire in a caſt-iron crucible; the heat was increaſed till the mixture in the crucible became perfectly fuſed, when it was poured out into an iron mortar, and ſuffered to grow cold and hard. It was of a dark green colour. After reducing it to powder, I poured upon it twelve pints of diſtilled water, with which I alſo rinſed out the crucible itſelf. The next morning I carefully decanted the clear liquor from the matter which had ſettled at the bottom, ſo, however, as to leave in the veſſel ſome of the liquor over the ſediment, which, after being ſhaken together, was poured out upon

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a filter of unſized paper: after the clear liquor had run from it, I lixiviated the sediment or reſiduum. ſeveral times, with freſh quantities of water, in order to extract all the ſaline matter.

All theſe liquors were ſubjected to evaporation in a glaſs matraſs, placed in a ſand-bath, the heat of which was ſuch as kept the liquor almoſt conſtantly boiling. The liquor was not put into the matraſs all at once, but at different times, a freſh quantity not being poured in till the preceding quantity was evaporated. The operation laſted ſeveral days. As early as the firſt or ſecond day there appeared cryſtals, which in figure, ſolidity, tranſparency, and other properties, reſembled vitriolated tartar, and were probably afforded by the potaſh.

Upon the dry reſiduum obtained by evaporation, I poured (not all at once, but different portions ſucceſſively, in order to prevent too violent an efferveſcence) double aqua fortis, as it is called, of which I found two pounds were requiſite for the complete ſaturation of the alkali, and

precipitation of all the metallic calx that was combined with it.

The precipitate, which was almoſt as white as milk, I poured out, together with the ſupernatant liquor, upon a filter of unſized paper. The liquor paſſed through clear, and yielded the next day cryſtals of nitre.

As I ſuſpected that a little nitre might be entangled in the white ſediment and be prevented from paſſing through the filter along with the liquor, I took care to edulcorate the reſiduum upon the filter ſeveral times with large quantities of boiling-water; after which I dried it, by expoſing it to the ſun for a whole day. I fancied, however, that I could ſtill perceive here and there ſome ſhining particles, which made me doubtful whether there might not yet be a little nitre remaining.

Experiment I. To two drams of this ſediment, I added a tea-ſpoonful of charcoal duſt, and with a very ſmall quantity of oil of rape-ſeed (after Mr. Hielm's method) I made the whole into a ſtiff paſte. I then braſqued a crucible, in the thick lining at

bottom

bottom of which, I made a pit or hollow for the reception of the paſte, which I put in after the lining was become quite dry, and ſet the crucible in the forge, applying a gentle heat till there ceaſed to ariſe either ſmoke or flame from the oil. The empty part of the crucible was now filled up with charcoal powder, and an intenſe heat was excited by means of double bellows, worked with a heavy load for the ſpace of an hour; after which, the crucible was taken out of the fire, and ſuffered to cool gradually in the warm aſhes.

When the crucible was become cold, I opened it, took out the contents, laid the portion that had been put in the before-mentioned pit or hollow, made in the lining, upon a piece of paper by itſelf, and waſhed off the charcoal that adhered to it.

The reſiduum weighed only two grains and an half, which conſiſted partly of black molecules, ſo little coherent as to crumble eaſily on being rubbed between the fingers; they were brittle, and without luſtre;—and partly of white opake globules, which were

probably derived from ſome fine particles of quartz that might have been left behind, and which with the fixed alkali had thus been melted to an imperfect glaſs.

Expoſed to the blow-pipe with glaſs of borax, the above-mentioned molecules, or agglutinated granules, gave an opake bluiſh-black ſpherule, that was not attracted by the magnet. Smoking ſpirit of ſalt poured upon them, acquired a yellow colour, which became deeper in the courſe of a few days. The greateſt part, however, remained undiſſolved, and appeared at the bottom of the phial without any change of colour: nevertheleſs there could be perceived amongſt theſe black granules, a fine yellowiſh white powder. On the addition of lixivium ſanguinis, or phlogiſticated alkali, the marine acid depoſited a blue ſediment; a proof that the black granules contained iron.

Experiment II. Suſpecting that the humidity which the ſediment uſed in the preceding experiment might have retained, had obſtructed its metallization, I roaſted or calcined another portion of ſediment in

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a ſtrong fire, whereby its bulk was conſiderably diminiſhed, though its ſpecific gravity was increaſed. Here and there it was of a yellow colour.

I put one drachm of this ſediment, mixed up with charcoal duſt and oil (as in Experiment I.) into a crucible ſimilarly braſqued, and expoſed it to the ſame intenſe degree of heat, kept up for the ſame length of time.

The reſiduum which was found in the crucible after cooling, I waſhed as before, and dried. It weighed ſeventeen grains and an half, and except a very ſmall part, was not attracted by the magnet. It conſiſted of pretty large hard agglutinated grains, which had more of a glaſſy than of a metallic luſtre, and were very brittle; neither theſe nor the other ſmall opake granules were fuſible before the blow-pipe, nor were they converted into a yellow powder. Expoſed to the blow-pipe with glaſs of borax, they gave an opake and bluiſh black globule. With ſmoking ſpirit of ſalt they ſhewed the ſame effects as in Experiment I. except

that in this caſe, yellow ſharp pointed cryſtals were produced.

The experiment proved as little ſucceſsful when performed, under ſimilar circumſtances in a potter's or porcelain furnace, though the crucible was ſuffered to remain ſeveral days in the hotteſt part of the furnace.

I repeated this experiment with two drams of the ſame kind of ſediment, in like manner mixed up (after having been thoroughly calcined) with charcoal-powder and oil, and put into a crucible ſimilarly braſqued, and provided with a lid that was tightly luted on. I ſent the crucible thus prepared to my friend Mr. Wille, at Beckerhagen, requeſting him to expoſe it for an hour or two to the hotteſt part of a forge, belonging to ſome iron-works of which he had the ſuperintendance. He obligingly complied with my requeſt, and ſent the crucible back to me, after having ſubjected it to the moſt violent heat of the forge for the ſpace of a couple of hours.

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The crucible was almoſt entirely covered over, both in the inſide and on the outſide, with a tranſparent green glaſs, in which there were many perfect metallic grains of different ſizes. Excepting only a few, they were attracted by the magnet, in which reſpect, as well as in their outward appearance, they reſembled iron; but internally, they exhibited a colour paler or whiter than that of iron; they broke into pieces with a very ſlight blow of the hammer *. Upon 15 grains of theſe metallic globules, I poured ſome ſmoking ſpirit of ſalt; a violent efferveſcence enſued, and a good deal was diſſolved. On the addition of a little more acid, a further diſſolution took place; but there was ſtill left a conſiderable portion undiſſolved.

* That Mr. Ruprecht was more ſucceſsful in his attempts to reduce tungſten or wolfram, is ſhewn by the account taken from Crell in the firſt vol. of our Journal, p. 38 (Some errata which occur in this account are corrected at the end of the Appendix) and alſo by a ſecond communication on the ſame ſubject, in the ſeventh No. of Crell's Annals, for 1790. In this laſt account Mr. Ruprecht relates, that the reguli of tungſten which he obtained, were of the ſize of ſmall ſhot. One of them expoſed under a muffle for half an hour to a white heat, in an aſſaying furnace, was not found to have undergone any alteration; it only left a blackiſh ſpot upon the cupel

WOLFRAM and ARSENIC.

Experiment III. Imagining that my want of ſucceſs in moſt of the preceding attempts was owing to the extraordinary refractorineſs

pel or ſaucer. Being afterwards heated to excandeſcence for a quarter of an hour, with glaſs of borax, it gradually melted, yielding a deep blood-coloured glaſs.—Another of the reguli ſuffered no alteration by digeſtion, under a boiling heat, with concentrated nitrous acid; nor was even ſtrong aqua regia, though aſſiſted by a boiling heat, capable of diſſolving either the regulus or its powder. Of its digeſtion with the pure vitriolic and muriatic acids no mention is made.—By repeated projection of flowers of ſulphur upon one of theſe reguli, heated to excandeſcence, its ſurface was turned blackiſh, and appeared to have undergone ſome degree of calcination. It left a black ſpot upon the capſule or ſaucer. (The ſpecific gravity of theſe reguli was found by Mr. Haidinger to be 6,823, whereas Meſſrs. d'Elhuyarts ſtate the ſpecific gravity of their reduced tungſten to have been 17,6.) This ſecond account of the proceſs, by which Mr. Ruprecht obtained the reguli above deſcribed, differs in ſome particulars, from that which was firſt publiſhed. In the firſt account it was ſtated (See the firſt vol. of this Journal, p. 40) that the mixtures were put into cavities or openings, made in the middle of pieces of charcoal, that were cut into a conical ſhape, and ſtuck or inſerted in ſmall crucibles, which ſmall crucibles were placed in a large crucible (filled to a third part with charcoal duſt) and were covered at top with a ſmooth flat piece of charcoal; and being thus diſpoſed, were ſurrounded with charcoal powder, with which alſo the remaining ſpace of the large crucible was filled to within an inch of the top; and laſtly, over the whole, was ſpread a layer of bone-aſhes or cupel-powder.——This was the mode of procedure that was firſt practiſed; but Mr. Ruprecht afterwards

of the wolframic metal, I had recourſe to the treatment which anſwered ſo well to Mr. Achard (Phyſico-chemical Works, vol. I. p. 238) in his experiments on platina; I

afterwards found it anſwer better to ſet aſide the excavated pieces of charcoal, through which the heat could not ſo readily penetrate, and to put one portion of the mixture (conſiſting of one part of tungſtenic calx, made into a thick paſte with an eighth part of charcoal-duſt and a little linſeed oil) at the bottom of a ſmall crucible, covered over with a thin piece of charcoal, made to fit exactly, and placed, ſurrounded with charcoal-powder, in a larger crucible;—the other portion of the mixture was applied to the inſide (immediately oppoſed to the tuyere or mouth of the forge) of the outer crucible; the remaining ſpace was filled up with charcoal-powder; and laſtly, over the whole was ſpread a layer, half an inch thick, of ſifted bone-aſhes. (The diſpoſition or arrangement of the mixture in the crucible, was not always alike. Thus, ſome accounts ſtate, that it was divided into three inſtead of two portions, that it was not laid at the bottom, but applied to one of the three ſides, and alſo to the oppoſite angle, &c. See the firſt vol. of this Journal, p. 284—306.) Thus arranged, the melting apparatus was placed upon a Hafnerzell brick, that ſerved as a pedeſtal, in a forge which was cloſed round with bricks of the ſame kind. As ſoon as the charcoal that was thrown in was thoroughly lighted, and the apparatus was ſufficiently heated, the double bellows with 80lbs. weight upon it, was ſet in motion, and continued to be worked for an hour and three quarters—(In Mr. R.'s firſt experiments the bellows were worked only an hour and a quarter; in other trials the time was an hour and a half, the weight upon the bellows being augmented towards the end of the operation; which laſt circumſtance, though not mentioned, is probably to be underſtood as having taken place, in the account from which this note is extracted)—during which time, freſh quantities of charcoal, ſprinkled with water, were conſtantly thrown in.

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mean the employment of *fixed* arsenic (i. e. white arsenic deprived of its volatility by fusion with potash) I was aware, indeed, that the more perfect metal platina is much more capable of bearing uninjured the action of the alkali which is contained in this preparation of arsenic; nor was I unmindful that the wolframic metal, in its imperfect or calciform state, attracts alkalis almost as strongly as acids do. Hence I had reason to fear that the calx of wolfram would ra-

—The heat thus excited, was so intense, that, after the operation, not only the pedestal, but also the upper part of the outer crucible, and even the bone ashes themselves, were found to be melted to a white spungy slag, hard enough to strike fire with steel—It was by means of this intense heat, with the treatment above described, that Mr. Ruprecht obtained what he considers to be regulus of tungsten. It is proper to add, however, that, Mr. Klaproth, of Berlin (Crell Ann. 1791, vol. I. p. 135—6) has repeated Mr. R.'s experiments without success. He exposed to a melting heat, in a *dute*, or assay-crucible, duly furnished with charcoal dust, some pure yellow acid of tungsten; to the same fire, and under the same circumstances, he also exposed some pure white calx of manganese. At the end of the operation, the manganese was found melted to a very fine regulus, but the acid of tungsten appeared in the form of a brownish-black powder, and had undergone no degree of fusion.

Note by the Editor.

ther unite with the alkali than with the inflammable principle or phlogiſton, i. e. would be converted into a neutral ſalt, inſtead of being reduced to a metallized or reguline ſtate. Nevertheleſs it was poſſible that the alkali might keep to the arſenical acid, and, combined with this acid, to which perhaps it has a greater affinity, might leave the wolframic calx untouched: And even though this calx ſhould attract the alkali more powerfully than the arſenical acid does, yet the interpoſed charcoal-powder might deſtroy the force of this attraction.

Accordingly I rubbed half a drachm of the ſame kind of wolframic ſediment as that which I had uſed in Experiment II, with an equal quantity of flowers or meal of arſenic, and of dry potaſh, together with a tea-ſpoonful of charcoal-duſt. This mixture I put into a crucible, lined, as in the preceding experiments, with charcoal-duſt, and expoſed it to the forge in the ſame manner as before, except that it was placed ſomewhat farther from the *form*, or tuyere.

At the beginning of the operation there aroſe arſenical fumes; the reſiduum in the

crucible

crucible after the operation, was more compact and less brittle than the residuum in the preceding experiments. It did not exceed eight grains, which consisted—of black molecules or globule (making up but a very small part of the whole)—of coherent black particles, and of a few granules and globules that had a dull drossy lustre: Very few of them had a metallic splendour; two or three resembled tin, and were probably derived from tin contained in the wolfram, and extracted from it by the potash.

Smoking spirit of salt contracted a yellow colour by digestion therewith, and yielded crystals of the same colour. Great part, however, of the black particles remained undissolved. On the addition of spring water, the marine acid became turbid; and on the addition of lixivium sanguinis or phlogisticated alkali, it deposited a blue sediment.

WOLFRAM and MANGANESE.

Experiment IV. Being desirous of reducing manganese, as well as wolfram, to a reguline state, and also of mixing them together by fusion,

fusion, I triturated one drachm of the same sort of wolframic sediment as was employed in Experiment I. with an equal quantity of Ilefield manganese; and with a tea-spoonful of charcoal-dust, and a little oil, I made the whole into a mass, which I put into a crucible in the manner before-mentioned, and placed in the forge. What remained in the crucible after the operation, weighed 18 grains. I could scarcely perceive in it any traces of fusion. There was here and there a globule which had more of a glassy than of a metallic lustre. There were, besides, a few coherent black grains; but the greater part of the residuum consisted of oblong, somewhat spherical, dark grey, opake, loose granules, which did not melt before the blow-pipe, and which, with glass of borax, yielded a deep violet-coloured opake globule.

Smoking spirit of salt poured upon these metallic grains acquired a yellow colour in the course of a few days. Still, however, there remained a good deal of black powder undissolved. On the addition of water, the acid became of a greenish colour;

and on the addition of Pruffian lixivium, or phlogifticated alkali, it let fall a blue precipitate intermixed with many fine white particles.

WOLFRAM and ZINC.

Experiment V. I next tried how far the more fufible metals were capable of promoting the reduction and fufion of this refractory metal, wolfram, and of uniting with it. I began with zinc. Knowing that the calx of the laft mentioned metal attracts the inflammable principle with great avidity, and is thereby revived, I mixed a dram and a half of the fame kind of wolframic fediment as was ufed in Experiment I. with an equal quantity of calcined zinc; and with the addition of a tea-fpoonful of charcoal duft and a little oil, I made the whole into a thick mafs, which I put into a crucible duly prepared, and kept in the forge for the fpace of an hour. Notwithftanding I had entirely filled up the empty fpace of the crucible with charcoal-powder, there arofe from it a flame, from which it was but too manifeft that

that part of the zinc was consumed as fast as it was revived; whence it was to be apprehended that very little of it would be capable of uniting with such a fixed and refractory metal as wolfram.

In fact, the greatest part of what was found in the crucible after the operation (it amounted to only 13 ½ grains) was burnt to a yellowish-white calx: The rest consisted of a black powder, that was attracted by the magnet, and of round particles and grains which had a dull lustre, and were not magnetic.

Smoking spirit of salt was scarcely coloured by these products, though they communicated to it a rough taste.

WOLFRAM and ANTIMONY.

Experiment VI. I triturated a drachm of the same kind of wolframic matter with an equal quantity of pulverized regulus of antimony, mixed them together with charcoal dust and oil, and treated the mixture in the same manner as in Experiment V.

The product consisted for the most part of a black powder, without any lustre, amounting to half a drachm; though it also contained some black agglutinated grains, which were very friable, and in which were intermixed a few shining globules, that resembled regulus of antimony. Only a small portion was attracted by the magnet. There were, moreover, some white semi-transparent globules, resembling those in Experiment I. and probably of a similar origin.

On the affusion of aqua regia bubbles were extricated. Water added to the acid, after it had stood over the metallic particles for some days, rendered it turbid, not indeed instantly, but in a short time.

WOLFRAM and BISMUTH.

Experiment VII. With bismuth the result was as little satisfactory. A drachm and a half of the wolframic sediment or calx, and an equal quantity of bismuth were treated in the manner before mentioned, except, that on account of the great fusibility of the bismuth,

muth, a leſs degree of heat was applied. The reſult, which weighed $42\frac{1}{2}$ grains, conſiſted of a black powder, among which were a few agglutinated particles, of a dark coppery luſtre. Some of them were attracted by the magnet.

Aqua fortis had little action upon them; at leaſt after ſtanding therewith for ſeveral days, it was not rendered turbid by common water.

WOLFRAM and COBALT.

Experiment VIII. My attempts to combine cobalt with this metal were not more ſucceſsful. From half a drachm of each, treated as in the preceding experiments, the product was for the moſt part, a black and greeniſh yellow powder, which, after waſhing and drying, weighed $46\frac{1}{2}$ grains. With this powder, however, there were alſo intermixed, ſome agglutinated particles and globules, which eaſily crumbled when rubbed between the fingers, into blackiſh brown grains. No metallic luſtre was perceivable.

Aqua regia poured upon the ſame, acquired a reddiſh colour, but a good deal of black powder was left undiſſolved.

By proper management I was in hopes I ſhould be able to bring the calx of wolfram to combine with the more perfect metals.—I began with lead.

WOLFRAM and LEAD.

Experiment IX. One drachm of lead, and an equal quantity of the ſame kind of wolframic calx as was employed in Experiment I. were treated in the manner already deſcribed; except that I did not apply ſo violent a degree of heat here, as I had done in the foregoing inſtances, on account of the great fuſibility of the lead, a property by means of which I was in hopes the lead would be able to bring this refractory metal into fuſion with itſelf. Nor was I diſappointed; for the mixture melted to a compact maſs, weighing two drachms. Nevertheleſs, excepting its outward appearance, which was altered by the charcoal-duſt that adhered to it, it very much reſembled, both in

in colour and fusibility, pure lead; and was, besides, nearly as soft as lead.

Experiment X. This result encouraged me to make another trial with the same kind of wolframic sediment or calx as was employed in Experiment H. Accordingly, I took half a drachm of this sediment, mixed it with half a tea-spoonful of charcoal dust, and wrapped it up in a thin plate or leaf of lead, weighing half a drachm, and put it into a crucible, proceeding in other respects in the same manner as before.

The result was somewhat different; for the residuum in the crucible, weighing forty-three grains, was not melted to a compact mass, but consisted—of very small globules, which resembled lead in appearance, but were rather harder;—of somewhat larger globules, which in their internal appearance resembled lead, but were harder, and some of them were brittle and spongy, like dross, in which however, globules of lead were perceivable;—of small bluish black particles, which for the most part were likewise brittle, and were probably prevented from melting

into globules of reguli, by a greater proportion of wolfram;—and lastly, of black powder,—of some particles that were attracted by the magnet,—and of a very few vitreous globules.

Aqua fortis poured upon the above-mentioned metallic globules, left great part of them undissolved.

WOLFRAM and TIN.

Experiment XI. I now proceeded to tin, whose effects or habitudes with this new metal I was the more anxious to learn, as I had reason to think that it (viz. wolfram) is present in most tin ores; that it is the cause of their remarkable specific gravity; and that in the smelting of the ore it may easily chance to mix and combine with the extracted tin. I therefore resolved to see in what manner tin would be affected by exposure to the fire along with wolfram.

Accordingly I took a drachm of the same sort of wolframic calx as was employed in Experiment I. mixed it with a tea-spoonful of charcoal dust, and having wrapped

ped it up in a drachm of tin-foil, laid it in the hollow made in the lining at the bottom of the crucible, and proceeded in other reſpects as in the preceding experiments, except that I did not, on account of the great fuſibility of the tin, apply ſuch a vehement heat.

After the crucible was become cold, I found that the contents had not melted into one compact maſs, but into a number of globules, amounting in the whole to twenty-ſeven grains and an half. They had the colour and luſtre of tin, except that the ſurfaces of ſome of them were irideſcent, or exhibited the colours of the rain bow, an appearance which is ſcarcely ever obſerved in tin that is perfectly pure. The other part of the product conſiſted of black powder and black molecules, which had here and there a dull metallic luſtre, and were compoſed of a congeries of grains, ſome of which were ſtrongly agglutinated, whilſt others of them were ſo little coherent, as to ſeparate and fall to pieces on the ſlighteſt preſſure of the finger. There was, more-

over, a ſmall number of minute vitreous globules, together with a few particles that were attracted by the magnet.

Aqua regia poured upon the above-mentioned agglutinated granules, became in a few days of a ſtraw colour; it was not rendered turbid by dilution with water; but on the addition of phlogiſticated alkali it let fall a greeniſh white powder. As to the reſt, a great part of the black globules and black powder remained undiſſolved.

Experiment XII. I repeated this experiment with half a drachm of that kind of wolframic calx that was uſed in Experiment II. wrapping it up (after having added charcoal duſt) in twice its weight (viz. one drachm) of tin-foil.

The reſult was ſomewhat different from that in the foregoing experiment; for the greater part of the tin was melted to a pretty large maſs or button, which weighed four ſcruples, and in colour and ſplendour reſembled tin, but from its much greater hardneſs, it evidently contained a foreign admixture or alloy. The reſt of the product

duct consisted of little globules (which constituted but a small part of the whole product); of a good deal of black powder; and of a few molecules, composed of agglutinated grains. Some particles were attracted by the magnet.

With aqua regia these metallic globules exhibited the same phenomena as those in the preceding experiment.

WOLFRAM and COPPER.

Experiment XIII. My next trials were with the less fusible metals. Of the same kind of wolframic matter as was used in Experiment I. I took a drachm, mixed it with an equal quantity of copper filings, added charcoal dust and oil, and, proceeding in other respects as before, put the crucible with its contained mixture into the forge, and subjected it to the most violent heat.

The product weighed forty-seven grains and an half, and appeared to have undergone only an incipient or imperfect fusion, for the shape of the smaller particles of the copper was but little altered, and the shape of the

larger

larger particles of the filings was not altered at all. Their colour, however, was altered; it now inclined to that of gold. Among the copper filings were obſerved a black powder, and ſmall black maſſes, ſome of which were pretty globular, and of a metallic luſtre. There were alſo ſome magnetic particles.

Aqua fortis diſſolved the greateſt part, and thereby acquired a greeniſh blue colour. There lay undiſſolved at the bottom of the veſſel, a ſmall quantity of black powder, which after ſeveral days appeared unaltered.

Experiment XIV. I repeated this experiment with half a drachm of the wolframic calx uſed in Experiment II. and an equal quantity of copper. The product, which weighed thirty-three grains, had evidently been fuſed; but the greateſt part of it conſiſted of ſmall looſe or diſtinct globules, which, in their external appearance reſembled pure copper; another part conſiſted of larger maſſes that ſeemed to be compoſed of agglutinated particles, cohering, however, ſo ſlightly as to crumble eaſily between the fingers:

fingers: among these were some that inclined to a blackish colour, which became still darker before the blow-pipe. In this case there was also a black powder, together with some particles that were attracted by the magnet.

With aqua fortis the effects were the same as in the preceding instance.

WOLFRAM and IRON.

Experiment XV. In order to see how iron would be affected by this new metal, I mixed a drachm of the same kind of wolframic matter as was employed in Experiment I. with an equal quantity of filings of crude or cast-iron, adding charcoal dust, &c. as before.

After the mixture had been exposed to the most violent heat of the forge, I found several different sorts of products. The greater part (four fifths of a drachm) which had scarcely begun to melt, and consequently had changed its form but little, was strongly attracted by the magnet, and differed from the filings before the experiment

only

only in inclining to a brown colour; A second part of the product, amounting to seventeen grains and an half, was melted into globules of different sizes, resembling tin in colour, splendour, and ductility, and likewise in not being attracted by the magnet; but they were somewhat harder than tin: A third portion consisted of black powder like that which was obtained in the other experiments: The fourth and least part consisted of small masses that seemed to be composed of minute agglutinated grains; they were black, had a very faint lustre, and no ductility. There were moreover, here and there attached to, and interspersed among these, some white metallic globules.

The unmagnetic portion imparted a fiery yellow-colour to smoking spirit of salt. There remained, however, at the bottom of the vessel a good deal of heavy black powder undissolved.

Experiment XVI. In order to determine from whence the tin proceeded that was obtained in the preceding experiment, I repeated

peated the ſame with the wolframic matter uſed in Experiment II. Of this I mixed half a drachm with an equal quantity of iron filings, adding charcoal duſt as before.

The reſult was very different. The whole was magnetic (i. e. was attracted by the magnet) and after ſome time contracted, all over, a ruſt, which ſhowed that the iron was equally diffuſed through the melted maſs. The ſhape and appearance of the filings was entirely changed. I could not perceive any veſtige of tin. Whence, then, proceeded the tin in the preceding experiment? Was ſome of the calx of tin (which is more ſtrongly attracted by alkali than moſt other metals) held in ſolution by the potaſh, and revived in the fuſion?

One portion of the product (about three fourths of a drachm) appeared in the form of a powder, or of very minute granules; another portion, which was of a greyiſh colour, was melted into little globular maſſes, which preſented a ſhining fracture; this luſtre, however, was more of a glaſſy than of a metallic kind. Their ſurfaces alſo exhibited

hibited the ſame luſtre when expoſed to the blow-pipe. Some of them were pretty large and hard.

Smoking ſpirit of ſalt poured upon theſe globules acquired a fiery yellow colour. A good deal of black powder remained undiſſolved.

WOLFRAM and SILVER.

Experiment XVII. Of the wolframic matter uſed in Experiment I. I mixed half a drachm with half a tea-ſpoonful of charcoal-duſt, and having wrapped it up in half a drachm of fine ſilver-leaf, I put it in the forge, applying a greater heat than was given to the mixtures with lead and tin, but a leſs heat than that to which the mixtures with copper and iron were ſubjected.

The ſilver was found caked or agglutinated with its incloſed ſubſtance, into a maſs that weighed a drachm; but neither its form, nor its colour, nor its luſtre, had undergone any change. The blackiſh, ſoft, ſpungy maſs incloſed within the ſilver, though it ſeemed at firſt ſight to be firm and compact,

proved

proved to be very incoherent and friable, and had neither assumed a metallic form, nor had in any part intimately combined with the silver.

Purified aqua fortis dissolved greatest part of it; but a good deal of black powder, together with some portions of the above mentioned spungy masses, remained undissolved.

Experiment XVIII. I repeated this experiment with half a drachm of the wolframic calx employed in Experiment II. and an equal quantity of silver-leaf. The result was very different. The whole was fused and weighed above a drachm. The silver was melted partly into irregular lumps, and partly into globules of various sizes. A portion of these globules were imbedded (so however, as to be easily distinguished by their colour and splendour) in black (and a few red) spungy, brittle opake lumps or masses, which had more the appearance of dross, than of glass or metal. Before the blow-pipe they melted pretty readily, so however, that the inherent grains of silver

remained

remained unaltered. Amongst these there was a good deal of black powder, many particles of which were attracted by the magnet.

With aqua fortis the effects were the same as in the preceding experiment.

WOLFRAM and GOLD.

Experiment XIX. Of the wolframic substance used in Experiment I. I mixed seven grains and an half with an equal quantity of gold, and after adding a little charcoal dust and oil, I proceeded as in the foregoing experiment.

The mixture was melted into a lump or button, that weighed ten grains and an half. It was still of a gold colour; it was, however, not quite so soft and ductile as gold, but exhibited cracks after several strokes of the hammer. Besides this button, there were also opake, grayish-white masses, which resembled dross, and seemed to be composed of agglutinated globules: there were also a few brittle black granules and particles.

Upon

Upon the laſt mentioned particles aqua regia had little action; but it turned the black powder white. It diſſolved moſt of that portion of the yellow button which I threw into it, and thereby acquired the uſual bright yellow colour: it left, however, undiſſolved a whitiſh piece of nearly the ſame ſize.

Notwithſtanding all the Experiments of the Spaniſh and German Chemiſts (not to mention thoſe of Scheele) on Wolfram, the ſubject is ſtill by no means exhauſted. As it occurs plentifully enough in the tin-mines of Cornwall, it is to be hoped that its properties and affinities will be further inveſtigated by ſome of our own chemiſts—There is reaſon to ſuſpect that ſome of Mr. Gmelin's calx or acid of wolfram (the ſediment uſed in Experiment I.) was not quite pure. As crude wolfram contains a variety of ingredients (See Keir's Dictionary of Chemiſtry, p. 148) great care ſhould be taken, in preparing its acid or calx, to free it from all heterogeneous matter.

Note by the Editor.

XXXI. EXPERIMENTS ON

MOLYBDÆNA,

WITH

A View to its Reduction.

By Mr. HIELM.

(*CONCLUDED*)

§. XXXIV.

IT has been already mentioned in a former part of this dissertation (§ XXIX. p. 242) that the readiest way of ascertaining whether the calx of molybdæna, prepared for experiments, be pure and free from vitriolic acid, is to melt it with iron. If the calx contain any of the aforesaid acid, the iron becomes red-short, and melts before the blow-pipe with intumescence, accompanied with a projection of sparks; and generally too it gives out an hepatic smell, on the affusion of acids.

By

By means of this criterion Mr. Hielm found that the calx of molybdæna which he had prepared according to M. Pelletier's method (§. XXIII, p. 225) was not entirely free from vitriolic acid*.

As neither Scheele's nor Pelletier's method could be depended upon *ſingly*, recourſe was had to the employment of them (agreeably to the remark made in §. XXVI. p. 222.) *conjunctly*.

Accordingly, calx of molybdæna prepared by ſublimation, was made into a ſoft paſte with nitrous acid, which was afterwards expelled by a ſtrong heat. After the calx was become cold, a freſh quantity of nitrous acid was again added to it, and was again evaporated. This was repeated four or five

* By extraneous admixture from which the calx of molybdæna, prepared after Mr, Pelletier's manner, is ſaid to be free, in a former part of this treatiſe (§. XXVI. p. 226) is to be underſtood earthy matter, ſulphur, iron, &c. i. e. the matrix *(bergart)* and mineraliſing matter. But that ſome vitriolic acid, formed, during the calcination, by the decompoſition of the ſulphur, may be carried up along with the ſublimed calx or flowers, it is eaſy to conceive. Hence the neceſſity of ſubjecting the calx thus obtained to a further purifying proceſs.

Tranſlator.

times; after which the calx was expoſed to a white heat. It was then taken out of the crucible, and put into a matraſs, and boiled for a quarter of an hour with diſtilled water. After the third boiling (freſh diſtilled water being uſed each time) the filtrated liquor ſhewed no trace of vitriolic acid, on adding to it ſome drops of a ſolution of terra ponderoſa in marine acid. The boiling was repeated, with freſh quantities of water, a few times more till the liquor, inſtead of a yellow, left a bluiſh white fine powder, on being evaporated upon a flat piece of glaſs. The calx of molybdæna was then ſeparated from the liquor by filtration, was edulcorated ſeveral times with hot water, and dried; and laſtly was expoſed to a red heat. By this treatment the calx was obtained free from vitriolic acid.

§. XXXV.

CALX of MOLYBDÆNA and NICKEL.

Nickel was depurated and reduced according to Bergmann's directions. The obtained regulus was but ſlightly attracted by the

the magnet. It was neither perfectly brittle, nor yet semi-ductile—The assay (i. e. the nickel and molybdænic calx) was put into a crucible upon a bed of charcoal, and covered over with charcoal powder. The arrangement in the forge was the same as before.

(a) Nickel four pounds, molybdænic calx one pound and an half.

After half an hour's exposure to the blast, a large button or regulus was obtained, that weighed three pounds and was not attracted by the magnet; and also several small globules that were readily attracted by the magnet. Before the blow-pipe these globules melted with difficulty, even on the addition of borax. Internally the regulus or button was of a dark grey colour; externally of a reddish yellow; it split under the hammer. By calcination it became yellow; and on the affusion of acids an hepatic smell was perceived. This last effect Mr. Hielm ascribes to vitriolic acid present in the nickel.

(b) Nickel four pounds, molybdænic calx two pounds, gave a button that weighed five pounds, and was difficultly fusible

per ſe, but melted readily with borax. In other reſpects like the preceding.

(c) Nickel four pounds, molybdænic calx four pounds. Button flat; grey internally; weighed ſeven pounds. In other reſpects like the preceding.

§. XXXVI.

CALX of MOLYBDÆNA, and REGULUS of COBALT.

(a) Pure regulus of cobalt four pounds, molybdænic calx half a pound, melted to ſmall globules, that were magnetic; difficultly fuſible both per ſe and with borax, to which, however, they communicated a fine blue colour. Did not betray any hepatic ſmell on the affuſion of acids.

(b) Cobalt four pounds, molybdænic calx two pounds. A flat oblong button, weighing five pounds and an half; melted with difficulty per ſe.

(c) Cobalt four pounds, molybdænic calx four pounds. An oblong button weighing ſix pounds and an half; externally black, internally white and porous.

§. XXXVII.

CALX of MOLYBDÆNA and REGULUS of ANTIMONY.

(a) Pure regulus of antimony eight lbs. molybdænic calx half a pound. Though the fuſion was performed in a well cloſed crucible, yet the whole was diſſipated. By means of a magnifying glaſs ſmall metallic grains were perceived on the ſides of the upper inverted crucible.

(b) Antimony eight lbs. molybdænic calx one lb. gave a ſpungy lump or maſs, which weighed three pounds, and had a metallic luſtre. It was not fuſible per ſe, but the antimony eliquated in a metallic form. It was ſcarcely ſoluble in borax. In the obtained glaſs, reguli of antimony were perceiveable.

(c) Antimony eight lbs. molybdænic calx two lbs. Product, which reſembled the preceding, weighed three lbs. Neither this nor the other gave out an hepatic ſmell on the affuſion of acids.

§. XXXVIII.

CALX of MOLYBDÆNA and ZINC.

(a) Pure regulus of zinc eight lbs. molybdænic calx half a pound. Diſſipated or exhaled in the ſame manner as the mixture, §. XXXVII. (a).

(b) Zinc eight lbs. molybdænic calx one lb. gave a greyiſh metalline maſs, that was brittle, infuſible per ſe, did not burn with a flame, and was difficultly ſoluble in borax.

(c) Zinc eight lbs. molybdænic calx two lbs. This mixture was diſſipated by ſublimation.

§. XXXIX.

CALX of MOLYBDÆNA and LEAD.

(a) Pure granulated lead eight lbs. molybdænic calx half a pound. One portion of the obtained button was ſoft, but nevertheleſs cracked at its edges on being hammered. The other portion was more brittle. Expoſed upon a piece of charcoal to the blowpipe, both portions exhaled molybdænic

calx

calx, and left not only a yellow colour (which proceeded from the lead) but alſo a bluiſh pellicle upon the charcoal. Much of this metallic compound or alloy was converted into droſs during its fuſion.

(b) Lead eight lbs. molybdænic calx one lb. Product, a yellowiſh grey, metallic, brittle maſs, that was difficultly ſoluble in borax, and gave no perfect button.

(c) Lead eight lbs. molybdænic calx two lbs. This product reſembled the preceding.

§. XL.

CALX of MOLYBDÆNA and BISMUTH.

(a) Pure regulus of biſmuth eight lbs., molybdænic calx half a pound. Sublimed into the upper crucible, adhering to the ſides of which were found a number of little grains.

(b) Biſmuth eight lbs. molybdænic calx one lb. Gave a light grey, metallic, thin plate, from which the biſmuth eliquated. It was difficultly ſoluble in borax.

(c) Biſmuth

(c) Biſmuth eight lbs. molybdænic calx two lbs. Product weighed three lbs. and reſembled the preceding.

§. XLI.

CALX of MOLYBDÆNA and TIN.

(a) Pure tin-filings eight lbs. molybdænic calx half a pound. Product malleable, but cracked on the edges after ſome ſtrokes of the hammer, and yielded a conſiderable quantity of droſs during its fuſion. Afforded a large button, together with ſeveral ſmall globules.

(b) Tin eight lbs. molybdænic calx one lb. a flat porous maſs weighing nine lbs. Gave both by itſelf, and with borax, a regulus of tin.

(c) Tin eight lbs. molybdænic calx two lbs. A ſpungy metallic maſs or lump that weighed eight lbs. was harder than iron, and ſcarcely ſemi-ductile. In other reſpects like the preceding. This compoſition was the moſt metallic of all.

§. XLII.

§. XLII.

CALX of MOLYBDÆNA, and REGULUS of ARSENIC.

The regulus of arſenic here made uſe of, was prepared from white arſenic firſt ſublimed, and afterwards reduced with black flux. It was ſlightly attracted by the magnet.

(a) Regulus of arſenic eight pounds, molybdænic calx half a pound. Melted into globules of different ſizes, that were attracted by the magnet. Subjected to ſcorification they melted into pure reguli, which projected ſparks and exhaled an arſenical ſmell. An hepatic ſmell was ſlightly perceivable.

(b) Arſenic eight pounds, molybdænic calx one pound, yielded a brittle regulus, in moſt reſpects ſimilar to the preceding.

(c) Arſenic eight pounds, molybdænic calx two pounds. The obtained regulus or button, which was brittle and in other reſpects like the foregoing, weighed eight pounds and an half. The preſence of vitriolic acid in this arſenical regulus was ſufficiently evident.

By

By ſolution in pure nitrous acid all theſe compoſitions or alloys were decompoſable, whereby it was ſeen which metals were pure, which not. On digeſting the compound with antimony in this acid, the molybdænic calx remained at the bottom of the ſame ſhape as the piece that had been thrown in. It was perfectly white; a proof that both the ingredients of this compound or alloy had been uſed in a ſtate of purity. On the ſlighteſt agitation of the veſſel the molybdænic particles were ſeparated. The compound with lead was diſſolved in the ſame acid with a production of great heat, and an extrication of red vapours. There was left undiſſolved a brown powder, probably ochre or calx of iron, which the lead might have contained. That it contained vitriolic acid too, may be inferred from this circumſtance, that, together with a white ſhining powder, there alſo remained undiſſolved a quantity of black powder, which was regenerated molybdæna.

§. XLIII.

§. XLIII.

It occurred to Mr. Hielm, that by reviving molybdænic calx, and afterwards treating the obtained regulus with nitrous acid, any regenerated molybdæna that might be formed (in consequence of the presence of vitriolic acid) during the reduction, would be thereby separated, and the calx itself left behind in a state of purity.

Accordingly, calx of molybdæna prepared (after Pelletier's manner) by torrefaction, was revived by exposure to the most violent forge heat, for several hours. The specific gravity of the reduced mass was, at first, 4,056; but four days after was found to be 6,911. It was then pulverised, and digested with nitrous acid (small portions only being thrown into the acid at a time). After the boiling had been kept up for some time, there appeared a quantity of white powder, which settled at the bottom, and occasioned such a tremour of the retort, as made it necessary to take it off the fire. The liquor, which was poured off from the powder or sediment,

ſediment, was of a yellowiſh colour. After the affuſion of diſtilled water upon it, the powder was emptied out of the retort upon a paper filter, and further edulcorated with a freſh quantity of water. After the whole of the liquor (which filtrated ſlowly) had paſſed through, the aforeſaid white powder appeared blue. The water uſed in the edulcoration acquired a graſs green colour. Freſh quantities of diſtilled water were repeatedly poured upon the filter, and ſtill continued to be tinged of the ſame colour, whilſt the powder was evidently diminiſhed in conſequence of ſome of it being diſſolved—What remained upon the paper was now dried, put into a crucible, and gradually ſubjected to a red heat; after which the calx appeared perfectly white, and by edulcoration and drying was rendered highly beautiful and ſhining.

Having reaſon, however, to ſuſpect that it was not even yet perfectly pure, Mr. Hielm again revived it in a very violent and long continued heat. The obtained maſs was

was of a yellowiſh grey colour, and was much more porous and pipy than the preceding. It exhibited many ſmall metallic globules in its pores. The ſpecific gravity was found, at firſt, to be 5,625 and a few days afterwards 7,105.

This regulus was finely pulverized, and, being put into a glaſs matraſs, nitrous acid was gradually poured upon it. After the firſt effervеſcence was over, part of the powder ſettled at the bottom of the veſſel, and exhibited a beautiful blue colour. On a freſh addition of the acid this colour diſappeared. The mixture became conſtantly thicker and thicker; and on making it boil more ſtrongly, ſuch a ſpirting and agitation was excited, as made it neceſſary to take the matraſs from the fire. The mixture was then emptied out into a china-bowl, and evaporated to dryneſs in a ſand-bath. The reſiduum was of a blue colour. By repeated affuſion and abſtraction of freſh quantities of nitrous acid, the blue colour was rendered ſomewhat paler; but this effect at length ceaſing,

ceasing, the affusion of nitrous acid was discontinued. The powder was emptied out of the bowl into a large glass mortar, warm water was poured upon it, and after greatest part of the powder had settled at the bottom, the supernatant water, which was still thick and turbid, was poured off. By filtration it was rendered clear; it was of a green colour, and was set by for further experiments. The edulcoration was repeated till the powder in the water became white; on drying it, however, on the filter, the blue colour re-appeared. After further exsiccation in a crucible, it was again brought back to perfect whiteness. Two pounds of this powder and eight pounds of cast-iron being put (without any other addition) into a crucible, afterwards well closed, and subjected to a melting heat, the mixture did not melt to a button, but only yielded an irregular mass; whereas, a mixture of two pounds of the aforesaid powder, and eight pounds of iron filings, laid in a crucible brasqued with charcoal-dust, melted to a metallic button, which

which was ſtreaked and reticulated; it broke under the hammer, exhibiting in its fracture a ſomewhat foliated texture and bright grey colour. Expoſed upon charcoal to the blow-pipe, it melted with intumeſcence and with a projection of ſparks. But the product with caſt-iron neither entered into fuſion, nor gave out ſparks.—Though the molybdænic calx thus obtained was purer than any that had been before obtained, yet it was not brought to the deſired degree of perfection. It was therefore boiled with diſtilled water, dried and revived. Four pounds of the obtained regulus, brought into fuſion with eight pounds of iron-filings gave a white button that was friable, and for the moſt part melted to a black ſlag. It gave out a few ſparks during its fuſion. Thus, therefore, ſome progreſs in the purification of the calx was made, and hence there was reaſon to hope that by a further proſecution of the experiments, the object in view might be attained. But previous to giving an account of ſuch experiments, Mr. Hielm takes notice of the pro-

perties of the water employed in the edulcoration. Neither acids nor alkalis occasioned any precipitation. The water, whether after or before the addition of acids, being evaporated to dryneſs, yielded a yellowiſh green pellicle—On adding to it ſome ſolution of terra ponderoſa in muriatic acid, no precipitation enſued; but as ſoon as a little ſpirit of wine was dropped in, a precipitate was immediately let fall, which gave out an hepatic ſmell on expoſure to the blow-pipe, and was a true ponderous ſpar; a proof that the water contained vitriolic acid. The precipitated powder appeared of a yellowiſh grey colour in the water, but on expoſure to the blow-pipe became white. A little molybdænic calx might perhaps have been thrown down at the ſame time, ſo that the precipitate in this caſe conſiſted of a triple ſalt. But on account of its ſcanty proportion, it was not poſſible to obtain poſitive marks of the preſence of this calx. On the addition of Pruſſian lixivium or phlogiſticated alkali no precipitation at firſt took place; but after a ſhort time a brown powder

der fell to the bottom. The ſolution immediately ſhewed a re-action with acids, and effervesced on the addition of more Pruſſian alkali or lixivium ſanguinis. In the Pruſſian lixivium no acid was perceivable, but on the contrary a ſtrong exceſs of alkali. The aforeſaid brown precipitate was calx of molybdæna. From this examination it is evident that the water uſed in the edulcoration contained ſome vitriolic acid, together with molybdænic calx, and that this acid remains adherent to the calx, and renders it impure.

At the ſame time, however, the above examination ſhews, that the quantity of vitriolic acid that remains adherent to the calx is but ſmall. And as, in ſpite of all the pains taken to the contrary, many earthy particles were ſtill left in the molybdænic calx; it ſeemed probable that the adherent vitriolic acid was retained by theſe earthy impurities. Therefore, the laſt obtained regulus was calcined and afterwards diſſolved in cauſtic volatile alkali. The ſolution, which was readily effected, was of a blue colour, and

left undiſſolved a reſiduum which conſiſted of feldſpar, ſilex, and perhaps, too, a little gypſeous matter, all which had been preſent in the molybdænic calx. This ſolution was boiled in a retort, to which a receiver was adapted, till a white pellicle began to be depoſited all round on the ſides of the retort. The liquor was then poured out into a china bowl and evaporated to dryneſs. There remained behind a yellowiſh grey maſs, which ſeparated eaſily from the veſſel, and diſſolved entirely and readily in water, to which it communicated a blue colour. This ſolution was evaporated to dryneſs, and the reſiduum calcined in a crucible expoſed to a red heat. This molybdænic calx which was now become white, was boiled ſeveral times in diſtilled water. The ſolution which was of a bluiſh green colour, was filtrated and evaporated to dryneſs, and the reſiduous powder was ſubjected to a melting heat, by which the molybdænic calx was ſublimed in the form of flowers, which for the moſt part had a ſilver-luſtre. —The calx of molybdæna left upon the filter

filter was now of a white colour. It was ſubjected to reduction, whereby it acquired a yellower colour than the preceding; whereas the obtained reguli are whitiſh if there happen to be an admixture of vitriolic acid. The regulus in the preſent caſe exhibited a ſtrong metallic luſtre on being rubbed with the back of a knife, notwithſtanding that its fracture was dull. But as the laſt mentioned molybdænic calx ſtill ſhewed ſome veſtiges of volatile alkali, Mr. Hielm thought it probable that it alſo contained ſome vitriolic acid, which explains why the regulus itſelf was not ſo perfect as it ſhould have been. It was therefore, again calcined under a degree of heat, ſo moderate as not to cauſe any of it to fly off, and yet ſufficient for effecting a ſeparation of all that was required to be ſeparated. The water employed in edulcorating this calx contracted a bluiſh grey colour, which on the addition of more water became green, and by ſtill further dilution diſappeared, and left the water colourleſs. On evaporating it to dryneſs, there remained

behind a yellowiſh green pellicle. On adding to it ſome drops of muriated barytes, it became turbid, but no precipitation took place; and on diluting it with more water, the ſolution again became clear. This ſhows that the water did not contain any vitriolic acid. Neither did it ſhew any veſtige of volatile alkali; for after admixture with quicklime, it yielded no nitrous ammoniac, either on holding over it, or dipping into it a ſlip of paper moiſtened with nitrous acid. Hence it is evident that the molybdænic calx, which was now of a blue colour, was further depurated from thoſe impurities which had ſo obſtinately adhered to it. On melting it with iron-filings in a crucible lined with charcoal-duſt, it gave a brittle button; whoſe fuſion was accompanied with intumeſcence or ebullition, but not with ſparks. Expoſed upon a piece of charcoal to the blow-pipe it gave out fumes, entered into fuſion, and was completely abſorbed by the charcoal. In its reduction it loſt 25 per cent. in weight, and after expoſure to the blaſt for three quarters of an

hour

hour it appeared of a greenish yellow colour. Being kept in a state of fusion for the same length of time, it became of a dark grey colour, in some places of a light grey, with a metallic lustre. Its specific gravity was, at first, 7,000, and at last, after all the air had been expelled from its pores and interstices, 7,400, which may therefore be stated as the specific gravity of this metal relatively to water. In the outer flame of the blow-pipe this regulus is easily scorified, but in the inner flame this effect does not take place so readily. Digested with nitrous acid, it leaves behind a white shining calx, provided it be free from coaly matter.

All these properties afford, in Mr. Hielm's opinion, satisfactory characters of a pure regulus of molybdæna; whence he flatters himself that the extraction or reduction of this metal from its ore, can no longer be considered as impracticable or uncertain.

From the preceding experiments, we also see that the shortest, cheapest, and surest method of obtaining a pure calx of molybdæna, is, to roast the molybdæna in a cru-

cible and afterwards treat it with volatile alkali; for when either the volatile alkali or vitriolic acid is ſeparated, we may be ſure that the other aſſociate will alſo be diſengaged. By this mode the calx of molybdæna is procured free from admixture with any other earth, without whoſe ſeparation it would be difficult to bring it to a ſtate of perfect purity.

In the buſineſs of roaſting, care muſt be taken that the molybdæna be ſubjected only to a red-heat, that too great a quantity be not put in the ſame place at once, and that the whole be brought to an equal degree of fineneſs. To accompliſh this laſt circumſtance, Mr. Hielm had recourſe to the following method. In the largeſt crucible of the ſmalleſt ſets or neſts, is ſet the 4th crucible in decreaſing ſize, and into this are put an ounce and an half (*three loth*) of molybdæna well picked and cleanſed from its matrix and extraneous matter, and kept red hot for a couple of hours: It is then taken out of the fire, and, after cooling a little, is turned out upon an iron-plate and gently preſſed

or

or ground with a hammer. The fine powder is now ſeparated from the coarſer part by means of a hair ſieve. The coarſer part is put back again into the ſame crucible and roaſted as before, without any other covering than a looſe lid. The fine powder is put into a capſule or ſaucer of ſuch a ſize as will admit it to be fixed or ſuſpended in the middle of a crucible of the above-mentioned dimenſions. This crucible is then covered either with a looſe lid, or with an inverted crucible that reſts upon the edges of the capſule or ſaucer. The whole apparatus is then expoſed to ſuch a degree of heat as ſhall make the crucibles of ſomewhat a ſtronger red-heat than the contained capſule. After a couple of hours, the firſt crucible is taken out and the coarſer molybdænic particles are ſeparated by ſifting as before. The fine powder is either put into the capſule contained in the other crucible (in which the molybdænic calx is found to be brought to a yellowiſh white colour) or, is put into a freſh capſule fixed in

in a third crucible. The coarſe particles of molybdæna which remain after the ſecond ſifting, are put back again into the firſt crucible and roaſted as before; and this treatment is repeated 'till all the molybdæna paſſes through the ſieve, and its yellow colour is altered. It is then collected and levigated in a mortar. About eighty grains *(one-third loth)* are ſtrewed, ſo as to form a thin layer, at the bottom of the crucible, and an equal quantity is put into the capſule fixed or ſuſpended in the middle of the ſame crucible and covered over, as before, with an inverted crucible. By means of three crucibles arranged in this manner, like muffles, the molybdænic calx may be freed from it's impurities, and, without being liable to run together or evaporate, may be brought (more effectually than it can be under the muffle of an aſſay-furnace) to a white and ſhining powder, amounting (from the quantity above-mentioned) to one ounce *(two loth)* i. e. to two-thirds of the whole. The earthy matter and vitriolic acid which

ſtill

ſtill adhere to the calx, are to be ſeparated by treatment with volatile alkali in the manner before deſcribed.

Remark. The common nitrous acid, or aqua fortis, is known to contain an admixture of vitriolic acid, from which it is not thoroughly freed by precipitation (according to the uſual method) with ſilver, and re-diſtillation. For the ſeparation of the vitriolic acid, Mr. Hielm adds either muriated barytes (for contrary to what Mr. Sage has ſaid in the Journal de Phyſique for 1789, nitrated barytes will not anſwer) or pure vegetable alkali, to the aqua fortis. In the firſt caſe a regenerated ponderous ſpar is thrown down, and in the ſecond caſe we obtain cryſtals of vitriolated tartar.

After digeſtion in cauſtic volatile alkali, filtration, lixiviation, and exſiccation in a red heat, the reſiduum from the before-mentioned ounce of molybdænic calx amounted to one drachm *(a quarter loth)*. It was diſſolved in diſtilled water, evaporated to dryneſs, and the obtained reſiduum was expoſed in a capſule to a red-heat. The molybdænic

lybdænic calx, thus prepared, was revived in the manner before-mentioned, and the obtained regulus was ſubjected to the following experiments.—N. B. If the molybdænic calx, before its reduction, ſtill contain a little volatile alkali, it ſhould be triturated and roaſted over again: Or, it may be treated with nitrous acid, and afterwards expoſed to a red-heat, whereby the volatile alkali will be completely expelled.

§. XLIV.

Fuſion of Molybdænum (Revived Molybdæna) with other Metals.

1. PLATINA and MOLYBDÆNUM.

The platina was depurated in the manner before-mentioned (§. XXIX. p. 237). The crucibles were lined with charcoal-duſt moiſtened with diſtilled water; and after the mixture or aſſays were put in, the the crucibles were filled up with dry charcoal-powder. The crucibles were kept in the fire for half an hour, and the arrangement in the forge was the ſame as before.

(a) Platina half a pound, molybdænum half a pound, melted to a hard irregular maſs

or lump, that was of a close texture, of a light grey colour, and had a metallic lustre. It was brittle.

(b) This compound was bruised on an anvil and mixed with one pound of molybdænic calx. Fusion not more complete.

(c) Being again pounded and mixed with three pounds of platina, it would not melt to a round button; but a solution or combination had taken place, for all the platina particles had disappeared. The product was of a granulated texture, and full of blisters and cavities. It was of a bluish grey colour, hard and brittle. An excess of either ingredient renders the compound difficult of fusion, and this in proportion as the excess is greater. The proportion in (a) seems to be that which melts the easiest.—The product in (c) gave out an hepatic smell with acids, though none of the others did so; and as the compounds obtained in the fusion of molybdænum with the other metals, did not exhibit this phenomenon, Mr. Hielm began to suspect that it proceeded from the platina, whose surface only is affected by the purifying-method made use of. Half a

quintal

quintal, therefore, of platina was laid on a bed of charcoal-duſt, and expoſed to the forge for half an hour. The ſemi-fuſed or agglutinated platina was brittle, porous, and covered with a hard cruſt. On the affuſion of marine acid, hepatic air was extricated. Mr. H. does not recollect that this property has been noticed by any perſon before. If all platina poſſeſs this property, there can be little doubt, that, in its native ſtate, platina is mineralized. In order to free it from this impurity, it was diſſolved in aqua regia, and precipitated with ſal ammoniac. From the liquor which was ſtill browniſh, the brick-coloured precipitate was ſeparated by filtration; and after repeated edulcoration with hot water, it was dried, and kept red hot for an hour. It was now become a grey, heavy powder, which, however, ſtill contained traces of volatile alkali. It was therefore ſubjected for half an hour to fuſion per ſe, in the forge, whereby its particles were conglutinated into a maſs that was ſemi-ductile, and of a whitiſh colour. It combined by fuſion with an equal quantity of

of molybdænum; but the compound ſtill betrayed an hepatic ſmell; whence it appeared that this purified platina ſtill contained vitriolic acid, together with volatile alkali, which adhered to it with equal pertinacity. After fuſion on a bed of charcoal, the depurated platina ſtill gave out hepatic air. It was diſſolved in aqua regia, and the ſolution was evaporated to dryneſs. The reſiduum was kept in a red heat for ſeveral hours, and then ſubjected to fuſion, without additament, in the forge. The compound obtained by melting this platina with molybdænum ſtill gave out hepatic air, on the affuſion of marine acid, but much more ſparingly than before. The further proſecution of theſe experiments was deferred to a future opportunity; in the mean time, Mr. Hielm thinks that by melting it either with molybdænum, or molybdænic calx, and afterwards ſeparating it from the ſame, platina may be rendered malleable, and fit for being wrought into any kind of form.

§. XLV.

§. XLV.

2. GOLD and MOLYBDÆNUM.

Gold powder prepared in the uſual manner, and freed from iron and vitriolic acid, was here made uſe of.

(a) Gold four pounds, molybdænum two pounds, yielded a black brittle maſs that had loſt nothing in weight. On being re-melted, it became of a cloſer texture. On expoſure to the forge for half an hour, in a cloſed crucible, without charcoal-duſt, half a pound of the gold eliquated and lay in ſmall grains or reguli at the bottom of the crucible. Theſe reguli were malleable, did not ſuffer any change of colour by expoſure to a red-heat, but after fuſion on a bed of charcoal they became darker. A portion of them was ſcorified with borax; the glaſs remained tranſparent and colourleſs. The remaining maſs being melted in charcoal duſt gave a brittle compound, which melted with difficulty before the blow-pipe, during which the gold eliquated. It diſ-

ſolved

ſolved with difficulty in borax; the ſurface of the ſpherule was covered with granules of gold. After cooling, the glaſs appeared yellowiſh from the particles of gold that were ſeen through it. It was ſtrongly attacked by nitrous acid; and after boiling therewith, the gold appeared at the bottom of the veſſel, and over it was ſeen the pure white molybdænic calx.

(b) To two pounds of the above-mentioned melted compound maſs were added two pounds of gold. The fuſion was as imperfect as in the former caſe, and the product exhibited nearly the ſame phenomena.

(c) Equal parts of both metals yielded a brittle and yellowiſh black compound, which preſented nearly the ſame phenomena as the preceding.

(d) None of theſe compounds could be brought into more perfect fuſion in covered crucibles either with or without the addition of borax.

§. XLVI.

3. QUICKSILVER and MOLYBDÆNUM.

Purified quickſilver, made very hot in a china bowl, was triturated with molybdænum for an hour, heat being applied all the time; but no remarkable alteration took place. By ablution with water, the quickſilver recovered its former ſplendour and fluidity. Further attempts were made, under the application of a greater heat, &c. but no union could be effected. However, it is known that many other metals are incapable of amalgamation with quickſilver.

§. XLVII.

4. LEAD and MOLYBDÆNUM.

The lead was purified by fuſion with borax. It was afterwards finely filed.

(a) Lead three pounds and an half molybdænum one pound and an half, after fuſion in

in an open crucible weighed two pounds. (This loſs in weight muſt have been occaſioned by evaporation). The obtained maſs was of a light grey colour, and brittle.

(b) Melted with one pound of lead in a covered crucible, it ſhewed nearly the ſame properties as before.—The fuſible metal lead, had ſeparated from the molybdænum, and was divided in ſinking through the charcoal-powder into minute grains.

(c) To the preceding maſs were added two pounds more of lead, and the mixture was put into a crucible and covered over with a little charcoal-duſt. A lid was luted on the crucible. After expoſure to the forge, greateſt part of the maſs was diſſipated. The remaining aſh-coloured maſs exhibited before the blow-pipe the ſame phenomena as molybdænum; but aqua fortis diſcovered the preſence of calx of lead.

(d) Lead two pounds, molybdænum one pound, with the addition of a little charcoal, expoſed to fuſion in a covered crucible for half an hour in a wind-furnace, melted to a

black brittle maſs, which weighed three pounds, and to which a few malleable reguli of lead adhered.

(e) The laſt-mentioned maſs was mixed with eight pounds of lead, and put, without the addition of charcoal-duſt, into a crucible cloſed with a covercle, and melted for a quarter of an hour in the forge. The inſide of the crucible was covered over with a grey coating, and the covercle was beſet with fine metallic grains. The melted maſs weighed ſeven pounds and an half, was hard and ſomewhat malleable, and whiter than pure lead. Expoſed to fuſion on charcoal, it kept its form and was ſcorified on its ſurface. From a larger piece the lead eliquated, after great part of it had put on a ſcorified appearance. Hence it is evident that the lead had united with the molybdænum; and hence too it is alſo ſeen that the lead muſt be added in an over-proportion, in order to obtain a compound fit for uſe, and to prevent the whole from being diſſipated in fumes.

§. XLVIII.

§. XLVIII.

5. SILVER and MOLYBDÆNUM.

The ſilver was precipitated from its ſolution by copper, and uſed in its powdery form.

(a) From four pounds of ſilver, and two pounds of molybdænum no round button could be obtained. On repeating the fuſion in a covered crucible, without charcoal-duſt, two pounds of ſilver eliquated, whoſe ſurface was of a dull luſtre and white; and after expoſure to a red-heat, bluiſh. It was malleable, and its ſolution ſhewed ſome traces of molybdænum. In other reſpects, what has been ſaid concerning the fuſion with gold is applicable here. The remaining mixture, which conſiſted of nearly equal parts of both metals, being melted on a bed of charcoal-duſt, became ſomewhat more compact, but did not give a round button: It was brittle, and when broken exhibited a light grey colour, and granulated texture. On melting it per ſe, ſilver eliquated

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from it. Borax, though it diſſolved little of it, became rather of an opake white, and ſomewhat like horn; perhaps capable of being employed as an enamel. Nitrous acid diſſolved the ſilver, calcined the molybdænum, and left the white calx undiſſolved.

(b) Silver four pounds, molybdænum one pound, gave a malleable compound, which, however, did not melt to a round button: it was of a ſilver-colour and granulated texture. To glaſs of borax it communicated different colours according as it was held in the outer or inner flame of the blow-pipe, and according as it was kept therein a longer or ſhorter time. The blue colour which it gave, could not proceed from copper, becauſe the ſilver by itſelf did not tinge the borax of ſuch a colour.

(c) Silver one pound, molybdænum two pounds, melted to an uniform, granulated, brittle greyiſh lump. Expoſed on charcoal to the blow-pipe, the calx of molybdæna evaporated, and the ſilver remained behind on the ſurface of the charcoal. Borax was tinged by it of the ſame colour as it is by

the

the molybdænic calx, which was here present in excess.

(d) The above products were put together into a crucible, for the purpose of melting them into a button; but this could not be effected either in closed or open vessels, whether with or without the addition of borax. The introduced pieces were still of their former shape and brittleness; they only in some instances became blue on their undermost surfaces. The silver, therefore, does not separate in the fire from the molybdænum, unless calcination in the open air precede the fusion. By cupellation the separation is effected; but this happens more readily after previous calcination. This shews that it is no mineralized substance, but a real metal, that is here combined with the silver, rendering it difficultly fusible and brittle. This also applies to the products with gold and the other metals.

§. XLIX.

6. BISMUTH and MOLYBDÆNUM.

The bismuth was purified by fusion and afterwards reduced to powder.

(a) Bifmuth two pounds, molybdænum two pounds, after fufion in an open crucible weighed lefs than two pounds. The deficiency was owing to evaporation.

(b) The obtained mafs was black. brittle, and interfperfed with fmall metallic grains. On remelting it with one pound of bifmuth, the refult was the fame; greateft part of the bifmuth was evaporated. Some globules were found in the upper inverted crucible; but the greateft number at the bottom of the other.

(c) The mafs, which now weighed two pounds, was mixed with four pounds of bifmuth, and expofed to the fame degree of heat as before, but with a very fmall addition of charcoal-duft. This refiduum did not weigh more than the former ones, though the crucible was uninjured and well clofed.

(d) Four pounds of bifmuth, and two pounds of molybdænum, melted on a bed of charcoal, in a wind-furnace, gave a black brittle mafs, with a bifmuth-button, weighing altogether four pounds and three quarters. The button bore a few ftrokes of the hammer, but at length fplit into feveral

pieces

pieces. It was of a closer texture than bismuth alone, and by fusion some of it was scorified. It weighed one pound, and was very fusible. From the black mass no more regulus of bismuth was obtainable; glass of borax was tinged by it of the same colour as by molybdænum. By treating it with nitrous acid, a white powder was obtained, which consisted of molybdænic calx, and calx of bismuth.

(e) To the above product were added four pounds more of bismuth, and the mixture was exposed to the forge for half an hour in a crucible without charcoal dust. The obtained black brittle mass weighed two pounds, and on exposure upon charcoal to the blow-pipe, molybdænic calx was exhaled. There was a blue, a white, and a yellow deposition on the edges of the charcoal; and a black mass remained behind, which was not soluble in borax. The possibility of combining these two metals together is clearly shewn by experiment (d)

§. L.

§. L.

7. COPPER and MOLYBDÆNUM.

(a) Pure copper (*Streukupfer*) four pounds, molybdænum one pound and an half, gave an oblong maſs which was of a dull copper colour, and bore ſeveral ſtrokes of the hammer before it broke into pieces. It was paler than copper.

(b) Copper one pound, molybdænum two pounds, gave a reddiſh grey brittle maſs, which could not be rendered malleable by further fuſion. Expoſed on charcoal to the blow-pipe, it turned bluiſh, and gave with borax a red coloured glaſs. Nitrous acid diſſolved the copper and left behind molybdænic calx of the uſual colour.

(c) Copper and molybdænum, in equal quantities, melted to an oblong maſs that yielded to the hammer a little, but at length broke in pieces, exhibiting a granulated texture and a bluiſh colour mixed with red. It admitted of being filed; and the filed ſurface appeared paler than copper, and kept its luſtre a long time.

(d)

(d) On attempting to melt all the products together, the reſult was not more ſatiſfactory, than it was with the compounds of ſilver and gold. In this inſtance the obtained glaſs was tinged red, in conſequence of the ready ſcorification of the copper.

§. LI.

8. NICKEL and MOLYBDÆNUM.

(a) Pure regulus of nickel* and molybdænum

* Pure regulus of nickel was obtained in the following manner. Regulus of nickel, ſuch as is deſcribed at §. XXXV. p. 328, was roaſted for ſeveral hours under a muffle, and afterwards finely pulveriſed and digeſted in ſpirit of ſal ammoniac *for ſeveral months (mehrere monate)*; after which the ſolution was filtrated and evaporated to dryneſs. It left a white powder, which on expoſure to a red heat, was converted into a bluiſh maſs, and a yellowiſh grey powder. The blue part was cobalt. The yellowiſh part tinged borax of a hyacinthine colour.—The reſiduum left by the alkali, after being lixiviated, exſiccated in a red heat, and expoſed to the forge for a quarter of an hour, appeared green, and was in part attracted by the magnet. This green calx was roaſted and diſſolved in nitrous acid. A little iron was left undiſſolved. The ſolution was of a beautiful dark green colour. Being evaporated to dryneſs, it left behind a greeniſh powder, which was roaſted for four hours, and then reduced, with charcoal powder, to a regulus, that was not wholly magnetic, bore hammering a little, but broke into ſmall grains, externally white, internally greyiſh, and unmagnetic. Yet, notwithſtanding all the pains beſtowed on its purification, an hepatic ſmell was ſtill perceivable on the affuſion of acids.

dænum, taken in equal quantities, melted to an oblong button, that was externally and internally of a light grey colour; it yielded ſomewhat to the hammer before it broke, when it exhibited a granulated texture. It was not magnetic, and was not fuſible either per ſe or with borax. The compound had loſt nothing in weight.

(b) Nickel two pounds, molybdænum four pounds, though they did not melt together ſo well as the mixture in (a) were yet well united, and equal in weight to the introduced ingredients. In other reſpects this product reſembled the preceding. Both gave out an hepatic ſmell on the affuſion of acids.

§. LII.

9. ARSENIC and MOLYBDÆNUM.

(a) Equal parts of regulus of arſenic and molybdænum (mixed together with a little diſtilled water, which mode of mixing the ingredients together was alſo adopted in the preceding experiments) were put upon a bed of charcoal in a covered crucible, and

expoſed

exposed for half an hour in a wind furnace. The result was a black spungy mass weighing two pounds.

(b) After exposing this product, mixed with six pounds of regulus of arsenic, in the forge for half an hour, the inside of the crucible was found glazed. The residuous black porous mass weighed only one pound and an half, and had scarce any metallic lustre. On exposure to the blow-pipe it gave out fumes after the manner of molybdænic calx, but no arsenical smell. With borax it yielded a black glass, such as is obtained from an over proportion of molybdænic calx. With aqua fortis it produced effervescence and heat; a white powder was left undissolved, which on exposure upon charcoal to the blow-pipe, gave out no arsenical smell; neither did it melt, but remained in the form of a black spungy scoria, which gave to borax the same colour as calx of molybdænum. It is remarkable that the calx of arsenic is rendered so fixed by the molybdænic calx, that its presence ceases to be discoverable by its smell.

§. LIII.

§. LIII.

10. IRON and MOLYBDÆNUM.

(a) Clean iron filings two pounds and an half, molybdænum two pounds and an half melted to a round button, without loſs of weight. (The crucible was filled up with charcoal-duſt, and furniſhed with a luted cover). The ſurface of the obtained button was black and droſſy. It broke eaſily under the hammer, and yet was hard enough to make an impreſſion or indentation in the anvil. In its fracture it appeared of a bluiſh grey colour, and of a fine ſcaly and fine granulated texture, interſperſed with ſome ſhining ſpots. Before the blow-pipe it melted with intumeſcence, but without ſparks. It was difficultly ſoluble in marine acid, which, though aſſiſted with heat, left a reſiduum, which, however, ſoon diſappeared on the addition of nitrous acid. Diluted vitriolic acid had little action upon it. By precipitating, or by evaporating the ſolution, blue calx of molybdæna was obtained.

(b) Iron two pounds, molybdænum four pounds. Button not quite ſo perfect as

 the

the preceding; equal in weight to the introduced ingredients. Externally the button was black and drossy; it was magnetic and brittle: internally, it was fine grained, and of a light grey colour: before the blow-pipe it was not fusible per se, and dissolved with difficulty in borax.

(c) With cast-iron and molybdænum the products were similar to the preceding.

Of all metals, iron unites the readiest, and in greatest quantity with molybdænum; and there is reason to expect that this metal may serve as a medium for purifying and meliorating iron.

Equal parts of iron-filings and molybdænum exposed to the forge for half an hour in a closed, but unbrasqued crucible, did not melt to a round button, but yielded a brittle, granulated, bluish-grey metallic mass. This shows what a difference the brasque (or addition of charcoal powder) makes.

§. LIV.

11. COBALT and MOLYBDÆNUM.

By repeated fusions, a regulus of cobalt was obtained that neither obeyed the magnet,

net, nor gave out an hepatic ſmell on the affuſion of acids. It was therefore looked upon as pure.

(a) Regulus of cobalt two pounds, molybdænum two pounds, expoſed to the forge for half an hour, in a braſqued crucible, gave a well fuſed button, which, however, was not perfectly round; it was of a light grey colour, brittle, and weighed three pounds.

(b) This product was re-melted in the ſame manner with two pounds more of molybdænum. The obtained button, which weighed five pounds, was not yet round; externally it was of a ſparkling reddiſh grey colour; it was hard and brittle; unmagnetic; internally of a granulated texture, and of a dark bluiſh grey colour. It was not fuſible per ſe before the blow-pipe, but only became bluiſh on its ſurface. With borax it gave a blue glaſs. It was leſs fuſible than the former product (a). It was ſtrongly attacked by nitrous acid, though a boiling heat was neceſſary to its complete diſſolution. White calx of molybdæna ſeparated from the red ſolution.

§. LIV.

§. LV.

12. TIN and MOLYBDÆNUM.

Tin purified from lead and other metals was melted with borax, and afterwards reduced to powder by filing.

(a) Tin-filings two pounds, molybdænum two pounds, melted to a blackiſh grey, granulated, brittle, ſoft maſs, weighing four pounds. The preſence of tin in this compound was not diſcoverable before the blowpipe either with or without borax. But on digeſtion with nitrous acid, calx of tin was found in the white powder, which, however, was not reducible on charcoal.

(b) Tin four pounds, molybdænum two pounds expoſed to fuſion in a braſqued and covered crucible, in a wind-furnace, gave a maſs equal in weight to the two ingredients, and ſomewhat harder than the former product, with which it agreed in other reſpects.

(c) The product in the laſt experiment was mixed with four pounds of tin and put in the forge for a quarter of an hour. The

obtained maſs weighed eight pounds, and was for the moſt part of an uniform texture. In this caſe no charcoal-duſt had been added. This compound was harder than the preceding, admitted of being hammered a little, did not crackle like tin when bent backwards and forwards, broke in two, and exhibited in its fracture a greyiſh colour, and granulated texture.

(d) This compound, whoſe ſurface was reddiſh, was re-melted in charcoal-duſt. Though the crucible was well cloſed, the charcoal-duſt was conſumed. The obtained button was covered with a black ſcoria, and weighed as much as, or rather more than, it did before the experiment. It bore hammering a little. Internally it was of an aſh-grey colour and granulated texture. Before the blow-pipe its ſurface calcined; the tin did not eliquate till the maſs was preſſed with the forceps. The tin was reduced to a calx by fuſion with borax: but unroaſted pieces of the alloy were not attacked by borax. Hence it appears that tin unites with molybdænum ſo as to form a compound,

whoſe

whoſe mixture is not deſtroyed in ſuch a melting heat as the above-mentioned.

§. LVI.

13. ZINC and MOLYBDÆNUM.

(a) Pure regulus of zinc (reduced to powder by filing) two pounds, molybdænum two pounds, were, with the addition of a little charcoal duſt, put into a covered crucible, and ſubjected to fuſion in a wind-furnace for a half an hour. The product was a black maſs, almoſt in a powdery ſtate.

(b) To this were added one pound of molybdænum, and ſix pounds of zinc, and the mixture was put (without the addition of charcoal-duſt) in a covered crucible, and kept in the forge for half an hour. A black ſpungy maſs was found at the bottom of the crucible. Viewed with a magnifying glaſs, it exhibited ſomething of a metallic luſtre in its fracture. Expoſed to the blow-pipe per ſe, it did not give out fumes; but by digeſting it in nitrous acid, a yellowiſh white powder was obtained, which volatilized on expoſure to the blow-pipe. The

 acid

acid had diſſolved the calx of zinc.—In the upper part of the crucible were ſeen a few metallic globules; and the lid and ſides were glazed by the vapours that had been forced up. This phenomenon happened in all the experiments in which the admixed metals were volatile; but it was moſt ſtriking in the mixtures with arſenic.—The volatility of the zinc prevented the mixture in the preſent inſtance, from melting to a perfect button.

§. LVII.

14. MANGANESE and MOLYBDÆNUM.

Manganeſe almoſt always contains an admixture of iron, or of vitriolic acid. From cryſtallized ore of manganeſe, Mr. H. prepared a few reguli of manganeſe that were free from the aforeſaid impurities.

(a) Equal parts of regulus of manganeſe and molybdænum melted, with the addition of charcoal duſt, to an irregular button, that was not fuſible per ſe, and did not colour glaſs of borax, till it had been roaſted. On

digeſting

digefting fome of it in aqua fortis, the molybdænic calx was left undiffolved. This is fufficient to fhow that thefe two metals are capable of being united.

§. LVIII.

15. ANTIMONY and MOLYBDÆNUM.

(a) Pure regulus of antimony* two pounds, molybdænum one pound, fubjected to fufion in the forge, gave a black, fpungy, incoherent mafs. The blow-pipe difcovered nothing but molybdænum in it; it fumed like calx of molybdæna, and after roafting tinged glafs of borax of the fame colour as calx of molybdæna does. It was ftrongly attacked by nitrous acid even in the cold; and on the application of heat, a white calx of both metals was obtained.

(b) Two pounds of each metal were fubjected to fufion with charcoal powder, in a

* Regulus of antimony was extracted from crude antimony, by fubjecting it to the fire with the addition of iron, and the regulus thus obtained was afterwards purified by ten different fufions with nitre. It was regulus of antimony thus purified that was ufed in §. xxxvii. p. 331.

wind-furnace. The inſide of the crucible became covered over with a white and greyiſh ſublimate. The reſiduum at the bottom of the crucible weighed ſomewhat leſs than two pounds, and in its properties reſembled the former product.

(c) To this were added ſix pounds of regulus of antimony, and the mixture, without the addition of charcoal-duſt, was ſubjected to fuſion in the forge for half an hour. The thin metallic maſs at the bottom of the crucible weighed one pound and an half, was externally of a yellowiſh grey colour, brittle; internally, it was bluiſh grey. It exhaled fumes on expoſure to the blow-pipe, but was not fuſible either per ſe or with the addition of borax. By digeſtion in acids, it was found to contain antimony: a yellow powder was obtained, which on expoſure to the blow-pipe on a piece of charcoal, gave out fumes, melted to a black ſcoria, and ſunk into the charcoal. Hence it appears that antimony is combinable with molybdænum.

From

From all these experiments the metallic nature of molybdænum is sufficiently apparent.—Numerous as the experiments here related are, Mr. Hielm assures us that many more have been made upon this metal in order to ascertain its properties thoroughly.—Though the other metals have been known for centuries past, yet fresh discoveries are continually made concerning their properties and uses; and many more still remain to be made. In the same manner, the uses and properties of molybdænum are only to be learned by time. When experiments are once made, it is easy for others to repeat and to vary them.

XXXII. CHEMICAL NEWS.

MR. PELLETIER's experiments on the aërated vegetable alkali (*Annales de Chimie, for October*, 1792) shew, that the alkali, however pure it may be, deposits an earth of a quartzous nature, on saturating it with fixed air; that, when perfectly saturated with fixed air, it crystallizes into rhomboidal tetraedral prisms terminating in diedral apices; that 100 grains of these crystals contain forty-three of fixed air, seventeen of water, and forty of pure alkali; that these crystals produce cold during their dissolution in water; and lastly, that in a very low degree of heat, the crystallized aërated alkali parts with some of its gas; but that all the fixed air cannot be expelled from it by heat alone.

According

According to Mr. Ribaucourt (*Ibid. November*, 1792) the following is the best receipt for making ink. Boil for an hour (or till half of the liquor is evaporated) in twelve pints of water,

Eight ounces of galls, and
Four ounces of logwood.

Strain the liquor through a linen bag, or hair-sieve, into an earthen vessel, and add to it,

Four ounces of green vitriol,
Three ounces of gum arabic,
One ounce of blue vitriol, and
One ounce of sugar candy.

The liquor must be stirred or shaken from time to time, to promote the solution of the ingredients, and especially of the gum; and when it appears that they are completely dissolved, the liquor must be left at rest for twenty-four hours. Then decant the ink from

from the grounds or sediment at the bottom of the vessel, and keep it in bottles well corked.

M. Pissis (*Ibid. for December*, 1792) recommends the employment of acetated barytes as a precipitant or re-agent for the examination of mineral waters. Aërated barytes reduced to powder is dissolved in distilled vinegar: the saturated solution has a bitter and roughish taste. It does not yield crystals on evaporation, but becomes gelatinous, like acetated magnesia. This gelatinous salt requires a considerable quantity of water for its dissolution. It decompounds or precipitates the following salts: vitriolated tartar, Glauber's salt, selenite, alum; nitre; sea-salt, sal ammoniac; mild or aërated vegetable alkali, and mild or aërated mineral alkali. It also throws down a precipitate from urine; whereas muriated barytes does not.—Now (according to Mr. P.) the presence of all vitriolated alkaline and earthy salts, except vitriolated tartar, and of all nitrated salts, except common nitre, may be

detected

detected by muriated barytes. After the water, therefore, has been examined by that precipitant, if acetated barytes be added it will show whether the water also contain vitriolated tartar, nitre, or any of the other salts that are not precipitable by the muriated barytes.

From Mr. Klaproth's analysis (*Journal de Physique, for October*, 1792, *from Crell, No.* 1, 1792) it appears that a quintal, of the red silver ore from the Hartz contains silver 60, regulus of antimony 20,3, sulphur 11,7, concentrated vitriolic acid eight parts. Another specimen from the neighbourhood of Freyberg, yielded per quintal, silver 62, crystallized antimony 18,5, sulphur 11, concentrated vitriolic acid, 8,5.

According to Mr. Sage (*Ibid. Nov.* 1792) the red silver ore from Peru contains per quintal silver 70, sulphur 18, arsenic 6, water and fixed air 6 parts; and the red silver ore from Alsace, silver, 7, sulphur 10, arsenic 78, water and fixed air 5 parts.

Mr. Westrumb (*Crell No.* 8, 1792) has detected the presence of sugar of lead in some

ſome ſnuff, from which he had often experienced head-ach, giddineſs, and ſometimes ſickneſs and cholicky pains. This ſnuff had been given him by a friend on his travels, and is ſuppoſed to have been purchaſed in Holland, whither it was probably imported either from France or Spain. Whether the ſnuff-makers mix ſugar of lead directly with their tobacco, or whether the tobacco moiſtened or ſeaſoned with vinegar, or any other vegetable acid, be put into veſſels made of lead, or lined with lead, are queſtions which Mr. W. leaves to be determined by others. In the mean while, it is of importance to the public to know that the ſnuff which produced the abovementioned diſagreeable effects, was found to be contaminated with lead.

We may ſhortly expect from ſome of the French chemiſts a ſet of experiments on the conſtituent parts of the carbonic acid, or fixed air, in which the experiments of our countrymen, Mr. Tennant and Dr. Pearſon will be confirmed. In particular, the deciſive experiment has been made of applying phoſphorus

phoſphorus to red hot lime, by which no charcoal is produced, but the phoſphoretum calcis (i. e. a compound of phoſphorus and lime) is formed, firſt diſcovered by Dr. Pearſon, which has the extraordinary property of ſetting water on fire.

Mr. Charles Hatchett, of Long-Acre, has lately had the honour of having a paper read in the Royal Society in which he deſcribes very accurately a new cryſtallization of ſpar from Derbyſhire, which he endeavours to ſhow is compoſed of the ſulphureous acid and lime, a compoſition heretofore unknown in the mineral kingdom.

A reſidue or depoſition from train oil, which has hitherto been of little value, and often thrown away as an impurity, has lately been uſed for making candles of a very excellent quality.

The whitening of linen and paper by means of the dephlogiſticated marine acid, anſwers the moſt ſanguine expectations of thoſe who have employed this acid for this purpoſe.

The

The Edinburgh college have not introduced into their new pharmacopœia ſo many improvements from chemiſtry as might have been expected. In the *Materia Medica* are inſerted *barytes* and *arſenicum*, but no preparations of them are given in the other part of the work. They have, however, inſerted the method of preparing the phoſphoric acid, and of making with it Dr. Pearſon's ſalt, the phoſphorated ſoda *(phoſphas ſodæ)*; and James's powder, under a title denoting its compoſition, according to Dr. Pearſon's analyſis, viz. *antimonium calcareo-phoſphoratum*. It is aſtoniſhing that neither the London nor Edinburgh college have introduced the *acid of tartar*, a ſalt ſo uſeful in the practice of phyſic.

The *ſal enixum* of the old chemiſts, the *acidulous ſulſat of potaſh* in the new nomenclature, the ſalt that remains in the retort after diſtilling nitrous acid from nitre and oil of vitriol, has been ſold by the Jews for acid of lemons. It has been propoſed to uſe this ſalt in its concrete form to allay

thirſt in fevers, and to increaſe the ſecretion in the mouth in caſes of parched and dry tongue. This acid ſalt is almoſt as palatable as the tartareous acid, and infinitely cheaper.

We underſtand that ſome practitioners here are making trials with the application of charcoal-powder to ſores and ulcers.

From Mr. Weſtring's further experiments (*Crell, No.* 5, 1792) on the uſes of the lichens in dying, it appears that among the *Lichenes imbricati*, the *L. luridus*, *L. centrifugus*, *L. ſaxatilis*, *L. parietinus*, *L. microphyllus*, *L. pulverulentus*, *L. omphalodes*, *L. diffuſus*, *L. fahlunenſis*, *L. ſtygius*, *L. phyſodes* are capable of giving to wool and ſilk various ſhades of brown, yellow, and grey, according as they are treated with lime and ſal ammoniac,—with common ſalt—with nitre—with vitriolated tartar—with alum—with fixed alkalis—with vinegar or other vegetable acid—with diluted marine acid—with ſolution of tin—with ſpirit of wine, &c.

&c. and according as they are ſubjected to maceration, digeſtion, or boiling—Mr. W. thinks that a fine red lake, or water colour might be prepared from the *L. parietinus.*

INDEX

THE INDEX TO THE THIRD VOLUME.

A.

B.

C.

E.

Fire,

CORRECTIONS.

Vol. I. p. 40, l. 4, for "*at one time with the white, at another with the black flux*" read "*at one time with alkaline phlogistic fluxes, at another with phlogistic substances alone.*"

Ibid. l. 17, for "*three parts full of*" read "*filled to a third part with.*"

Ibid. l. 18, dele "*some.*"

Ibid. l. 26, for "*about,*" read "*within.*"

Ibid. p. 41, l. 3, and 8, for "*potter*" read "*Hafnerzell.*"

Ibid. l. 4, and 8, for "*tile*" read "*brick.*"

Ibid. l. 8, for "*crooked*" read "*curved or semi-elliptical.*"

Ibid. p. 42, l. 4, for "*its,*" read "*their.*"

Ibid. p. 126, l. 8, for "*partly in a pure or unmixed state, and partly intermixed or stratified with, &c.*" read "*partly in compact lumps or unstratified, and partly interspersed or stratified through, &c.*"

Ibid. p. 194, l. 16, for "*salt water,*" read "*solution of salt in water.*"

Ibid. p. 198, l. 5, and p. 201, l. 2, for "*brandy*" read "*corn or malt spirits.*"

Vol. III. p. 84, after "*a heat of 50° or 60°* add "*of Reaumur's scale.*"

The lispound (which occurs so frequently in Mr. Hielm's paper) is equal to 20lbs.

Other errata are corrected at p. 206, 310, Vol. I.

At p. 112, 155, 156, 164, 215, Vol. II.

At p. 285, 287, 293, Vol. III.